Hexachloroxanthene Method Development Report

Focused Sediment Investigation Work Plan FSIWP

Lower Passaic River Study Area, Newark, New Jersey

Prepared for:
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East Brunswick, NJ

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ENVIRONMENTAL DATA SERVICES, LTD.

DATA VALIDATION • TECHNICAL WRITING
CONSULTING • DATA INTERPRETATION

Table of Contents

1. Purpose

1.1. Introduction

Collection and analysis of river sediment samples were planned as part of Tierra Solutions Inc.'s (Tierra's) Focused Sediment Investigation Work Plan, Quality Assurance Project Plan, Revision 1, December 2011 (FSIWP-QAPP). The planned analyses were designed to meet project data use objectives as defined in Worksheet # 10 of the FSIWP-QAPP, "Data generated as part of the focused sediment investigation will be used to further evaluate additional sources of 2,3,7,8-TCDD to the Lower Passaic River Study Area (LPRSA)." Laboratory results of sediment samples collected for the determination of hexachloroxanthene (HCX) were identified as one of the data types needed to fulfill the project data use objective as quoted above. In light of the defined data need to assess sediment sample concentrations of HCX, the technical project team developed a planned approach to meet the laboratory analysis objective. This report documents the development and assessment of the analytical approach used in the determination of HCX as implemented during the Focused Site Investigation.

1.2. Background

Initially a literature search was conducted to identify available current and/or historical analytical methods for possible use in the identification and quantitation of HCX. It was determined at the time the literature search was performed, that none of the US contract laboratories contacted were actively analyzing samples for HCX. However, two relatively recent historical references to applicable analytical methodologies for the determination of HCX in environmental matrices were identified and are listed below.

- Tracy, K.L., Misita, M., Schrock, M., and Tabor, J. 2003. Extraction and analysis of hexachloroxanthene using modified U.S. EPA Method 1613, Revision B procedures, *Organohalogen Compounds* Vol. 60, p.177-210
- Viswanathan, T.S., and Kleopfer, R.D. 1986. The presence of hexachloroxanthene at Missouri dioxin sites, in Rappe, C., Choudhary, G., and Keith, L.H., ed., *Chlorinated Dioxins and Dibenzofurans in Perspective*, Lewis Publishers, Inc, Chelsea, Michigan, p. 201-210.

2. General Description of Analytical Method

The existing analytical procedures described in the technical papers identified above were reviewed by Vista Analytical Laboratory (Vista). Based upon this review, method development work was conducted by Vista to reproduce and/or improve upon the analytical approach established in the historical work.

The analytical method developed by Vista for use in the identification and quantitation of HCX during implementation of the FSIWP is a modification of USEPA method 1668 "Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS." The stepwise procedure developed by Vista is fully described in standard operating procedure (SOP) L-1 revision 1, titled: Extraction and Analysis of Hexachloroxanthene by HRMS, Vista 2012, and is provided as Attachment A of this report.

HCX concentrations are determined by first extracting samples using methylene chloride. Sample extracts then undergo appropriate extract clean-up procedures and are analyzed via high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS).

As USEPA 1668 is a performance based method, the effectiveness of any modifications made to the base procedure must be thoroughly demonstrated and documented as such per the requirements specified in Section 9 of the procedure. Accordingly, the stepwise procedures outlined in SOP L-1, once developed fully, were subjected to the each of the performance evaluation studies outlined in Section 9 of USEPA method 1668. USEPA method performance criteria were met for the studies performed by Vista using the HCX SOP L-1 in all cases, prior to use in the FSIWP program.

3. Pre-Program Verification of Analytical Performance (Studies)

Once the stepwise analytical procedure (SOP L-1) was developed by Vista, the following quality assurance/quality control evaluations were performed for both solid and liquid sample matrices:

- Initial Precision and Recovery (IPR) Studies
- Method Detection Limit (MDL) Studies
- Method Blank Studies

Vista procured authentic and certified HCX reference material from Cambridge Isotope Laboratories. These certified reference materials were used to calibrate the HRMS as well as complete the required method performance studies. Each of the method performance studies are described the sub-sections below.

3.1. IPR Studies

3.1.1. IPR Study (Solid)

Vista performed a solid sample IPR study (See Attachment C) prior to sample collection activities. The results of this study meet all acceptance criteria as defined in USEPA method 1668 and are provided in Table 3.1.1 below.

Table 3.1.1
Results of Solid IPR Study

Analyte	Average Recovery %	Method Limits %	% RSD	Method Criteria % RSD
1,2,4,5,7,8-HxCX	99.0	60-140	4.7	40
^{13}C-1,2,3,7,8,9-HxCDF	96.3	35-135	2.4	50
^{13}C-1,2,3,7,8,9-HxCDD	98.3	35-135	1.7	50

RSD = Relative Standard Deviation

3.1.2. IPR Study (Liquid)

Vista performed a liquid sample IPR study (See Attachment D) prior to sample collection activities. The results of this study meet all acceptance criteria as defined in USEPA method 1668 and are provided in Table 3.1.2 below.

Table 3.1.2
Results of Liquid IPR Study

Analyte	Average Recovery %	Method Limits %	% RSD	Method Criteria % RSD
1,2,4,5,7,8-HxCX	75.4	60-140	9.5	40
^{13}C-1,2,3,7,8,9-HxCDF	92.9	35-135	4.9	50
^{13}C-1,2,3,7,8,9-HxCDD	103	35-135	2.9	50

RSD = Relative Standard Deviation

3.2. MDL Studies

3.2.1. MDL Study (Solid)

Vista performed a solid sample MDL study (See Attachment E) prior to sample collection activities. This MDL study was performed per the requirements specified in Part 136 of Title 40 - Protection of the Environment, Code of Federal Regulations (40 CFR), Appendix B. The results of this study are provided in Table 3.2.1 below.

Table 3.2.1
Results of Solid MDL Study

Analyte	Amount Spiked pg/g	Trial 1 pg/g	Trial 2 pg/g	Trial 3 pg/g	Trial 4 pg/g	Trial 5 pg/g	Trial 6 pg/g	Trial 7 pg/g	Average pg/g	MDL pg/g	PQL pg/g
Hexachloroxanthene	50	45.4	42.0	41.2	40.7	47.2	44.6	36.2	42.48	11.40	20.0

MDL = method detection limit
pg/g = picograms per gram
PQL = project quantitation limit

3.2.2. MDL Study (Liquid)

Vista performed a liquid sample MDL study (See Attachment F) prior to sample collection activities. This MDL study was performed per the requirements specified in Part 136 of 40 CFR, Appendix B. The results of this study are provided in Table 3.2.2 below.

Table 3.2.2
Results of Liquid MDL Study

Analyte	Amount Spiked pg/l	Trial 1 pg/l	Trial 2 pg/l	Trial 3 pg/l	Trial 4 pg/l	Trial 5 pg/l	Trial 6 pg/l	Trial 7 pg/l	Average pg/l	MDL pg/l	PQL pg/l
Hexachloroxanthene	500	287	307	300	289	286	315	294	296.86	34.59	50

MDL = method detection limit
pg/l = picograms per liter
PQL = project quantitation limit

3.3. Method Blank Studies

3.3.1. Method Blank Study (Solid)

Vista performed a solid sample method blank study prior to sample collection activities. The results of this study meet all acceptance criteria as defined in USEPA method 1668, and are provided in Table 3.3.1 below.

Table 3.3.1
Results of Solid Method Blank Study

Analyte	Control Blank Result pg/g	PQL pg/g
1,2,4,5,7,8-HCX	1.64 U	20

pg/g = picograms per gram
PQL = project quantitation limit
U = Not detected

3.3.2. Method Blank Study (Liquid)

Vista performed a liquid sample method blank study prior to sample collection activities. The results of this study meet all acceptance criteria as defined in USEPA method 1668, and are provided in Table 3.3.2 below.

Table 3.3.2
Results of Liquid Method Blank Study

Analyte	Control Blank Result pg/l	PQL pg/l
1,2,4,5,7,8-HCX	12.9 U	50

pg/l = picograms per liter
PQL = project quantitation limit
U = Not detected

4. Program Method Performance Criteria

4.1. Measurement Performance Criteria for HCX Data

Project measurement performance criteria were developed for the HCX data set and are provided in Worksheets #12, #15, #28-1 and #28-2 of the FSIWP-QAPP. Measurement performance criteria are used to assess data quality and thereby establish the reliability of sample results provided by the laboratory in meeting project data use objectives. Assessment criteria include acceptance limits for the following HCX analytical performance indicators:

- Precision
- Accuracy
- Representativeness
- Completeness
- Sensitivity

Additional key requirements regarding sample handling and holding time limitations which relate to the representativeness of the HCX results obtained for this study were developed and are outlined in the FSIWP-QAPP Worksheet #19.

4.2 Third Party Data Validation and Assessment

A data validation SOP was developed as a tool with which to objectively evaluate the quality of HCX data obtained during the Focused Sediment Investigation by a third party organization. SOP V-1 of the FSIWP-QAPP titled HCX Rev. 0, August, 2011 was used to perform the HCX data validation of sample results reported during the Focused Sediment Investigation. The data validation procedure is located in Appendix C of the FSIWP-QAPP. For convenience SOP V-1 has also been included as Attachment B of this report.

The data validation process provides a determination of the suitability of HCX results for use in the fulfillment of program objectives. This determination is based upon the criteria and guidance provided in SOP V-1, as well as assessment of the HCX analytical performance indicators outlined in Section 4.1 of this report, and overall adherence to the FSIWP-QAPP.

5. Conclusion

The method development process began by evaluation of analytical methods developed by others for the determination of HCX in environmental media. Noted successes and limitations of earlier work were considered as Vista developed a stepwise analytical procedure capable of identification and quantitation of HCX. USEPA Method 1668 was selected as the basis of the HCX procedure. The effectiveness of required modifications made to the base procedure were thoroughly demonstrated and documented as such via successful completion of the performance evaluation studies outlined in Section 9 of USEPA method 1668. USEPA method performance criteria were met for the studies performed by Vista using the HCX SOP L-1 in all cases, thereby demonstrating the ability of SOPL-1 to produce results for HCX that are of known and documented quality. Based upon the pre-program analytical method development work, HCX results obtained are expected to be of equal or better quality than those produced in previous studies by others.

The further establishment of method performance criteria for HCX in the FSIWP-QAPP, and the assessment of achieved performance through implementation of data validation procedures by a third party organization, serves to establish the level of data quality achieved.

Attachment A

Vista Analytical SOP

SOP L-1 Revision 1, Extraction and Analysis of Hexachloroxanthene by HRMS, Vista 2012

SOP 41	Revision: 1	Supersedes: R0
EXTRACTION AND ANALYSIS OF HEXACHLOROXANTHENE BY HRMS		
Analyst Review: Melanie Schuld		
Management: Madhu Moier		
Quality Assurance: Ron Hamilton		
Effective Date: 16 April 2012		

Revision	Description of Revision

1. PURPOSE

1.1. This procedure describes the preparation and analytical techniques used for the analysis of soil/sediment and aqueous samples for the determination of Hexachloroxanthene (HCX) by HRMS.

2. SUMMARY OF METHOD

2.1. This procedure uses matrix specific extraction, analyte specific cleanup, and HRGC/HRMS analysis techniques.

2.2. This method covers the determination of Hexachloroxanthene (HCX). The following parameter may be determined by this method:

Parameter	CAS No.
1,2,4,5,7,8-Hexachloroxanthene	38178-99-3

2.3. Detection limits are sample-specific.

3. INTERFERENCES

3.1. Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferants under the conditions of analysis by performing laboratory method blanks. Analysts should avoid using PVC gloves.

3.2. The use of high purity reagents and solvents helps minimize interference problems.

3.3. Interferants co-extracted from the sample will vary considerably from matrix to matrix.

3.4. If interferences are encountered, the method provides selected cleanup procedures to aid the analyst in their elimination.

4. DEFINITIONS

4.1. Definitions are presented in the Glossary.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Vista employees, including the appropriate use of Personal Protective Equipment and engineering controls.

5.2. Each chemical compound should be treated as a potential health

hazard. Exposure to these compounds should be reduced to the lowest possible level. Only highly trained personnel thoroughly familiar with handling and cautionary procedures and the associated risks should handle all compounds or reagents.

5.3. Each chemical compound should be handled in well-ventilated, controlled access laboratories.

5.4. Additional health and safety information can be obtained from material safety data sheets (MSDSs) available to all personnel involved in these analyses.

5.5. In the event of a known or potential compromise to the health and safety of a Vista associate, all work must stop and the incident reported immediately to management.

6. APPARATUS AND MATERIALS

6.1. Analytical Balances, capable of reading to 0.01g and 0.0001 g

6.2. Crimp top autoinjector vials plus caps and crimp tool

6.3. Drying Oven, VWR Model 1320 or equivalent

6.4. Electrothermal electromantle six sample and 500 & 1000 mL capacity

6.5. Funnels, 100 mm

6.6. Glass columns, 160 mm x 11 mm and 200 mm x 15 mm

6.7. Glass wool

6.8. Organomation 24-Station N-Evaporator

6.9. Precleaned Glass fiber thimbles - coarse

6.10. Rotary evaporator

6.11. Round bottom flasks: 50, 100, 250, and 500 mL

6.12. Separatory funnels, typically 250 mL to 2-L size

6.13. Soxhlet Extractor

6.14. Teflon boiling chips

6.15. Test tubes plus Teflon lined caps, 16 mm x 125 mm

6.16. Vials, Glass conical,

6.17. Volatile Organic Analysis (VOA) vials, 40 mL

6.18. Wiretrol II Precision Disposable Micropipettes

6.19. Zymark TurboVap II plus 250 mL tubes with 1 mL stems or equivalent

6.20. EquipmentCTC Autosampler Model A200S.

6.21. Alpha Station 500.

6.22. Neslab HX200, HX300 or HX500 Water Cooler.

6.23. HP 6890F Gas Chromatograph

6.24. 60 meter DB-5MS GC column, 0.25 mm i.d., 0.25 μm film (J&W Scientific) or equivalent

6.25. Waters Autospec Ultima Magnetic Sector High Resolution Mass Spectrometer.

6.26. Injection vial inserts, 100 μL (Sun International or equivalent)

7. REAGENTS, SOLVENTS AND STANDARDS

7.1. Reagents (Highest purity available)

7.1.1. Acid Silica Gel, 44%

7.1.2. Activated Silica Gel, kilned for ~5 hours at 550°C, granular

7.1.3. Anhydrous sodium sulfate, kilned for ~5 hours at 550°C, granular

7.1.4. Basic Silica Gel, 33%

7.1.5. Florisil

7.1.6. Acid Alumina

7.1.7. Hydrochloric acid, concentrated

7.1.8. Hydromatrix

7.1.9. Ottawa sand, kilned for ~5 hours at 550°C

7.1.10. Ultra-pure nitrogen gas

7.1.11. Water, HPLC grade

7.2. Solvents

7.2.1. Tetradecane

7.2.2. Hexane

7.2.3. Methylene chloride (DCM)

7.2.4. Toluene

7.2.5. Acetone

7.3. Standards

7.3.1. All analytical standards are obtained from a certified vendor. See SOP 15 and the current spike sheet for more information.

8. QUALITY CONTROL

8.1. Method Blank (MB): Method blank is a matrix preparation that is free of native analyte that has been prepared and analyzed using the same procedures followed for the rest of the analytical batch. The method blank should simulate (as close as possible) the matrix to be extracted.

8.1.1. A method blank is run with every analytical batch or 20 samples (whichever is less).

8.1.2. For the determination of the native Hexachloroxanthene, the levels measured in the method blank must be less than the method quantitation limit or ten times lower than the concentration found in any sample within the analytical batch.

8.1.3. All samples within an analytical batch are re-extracted and analyzed if the method blank associated with that batch does not meet criteria.

8.2. Ongoing Precision and Recovery (OPR): An OPR is prepared by adding a known quantity of native standards to an interferant free matrix and used to assess method performance (precision and accuracy).

8.2.1. A 10 µL aliquot containing 4ng HCX is used for spiking.

8.2.2. The control limits are 50-150% for HCX.

8.2.3. If the OPR of an isomer is outside the recommended control than the sample and the OPR will be re-extracted and analyzed.

8.3. Matrix Spike (MS/MSD): Upon client request, a matrix spike sample is prepared by adding a known quantity of native standards to a sample matrix prior to extraction.

8.3.1. An MS/MSD is performed upon client request.

8.3.2. A 10 µL aliquot containing 4ng HCX is used for spiking.

8.3.3. The relative percent difference between MS/MSD samples should be 20%.

8.4. Duplicate Samples: Duplicate samples are two separate aliquots taken from the same source. Duplicate samples are performed upon client request.

8.4.1. A duplicate sample is performed upon client request.

8.4.2. Duplicate samples are analyzed independently to assess laboratory precision.

8.4.3. The relative percent difference from duplicate sample analyses should be less than 25%.

9. COLLECTION, PRESERVATION, AND HANDLING

9.1. Extract aqueous samples within 14 days and sediments within 14 days. Analyze within 40 days from extraction

9.2. Store all samples at 4°C in the dark

9.3. If residual chlorine is detected in an aqueous sample, add 80 mg sodium thiosulfate per liter.

10. SAMPLE PREPARATION

10.1. Residual Chlorine Determination (aqueous only)

10.1.1. Obtain an Aquacheck strip and place it directly into a small amount of sample in a disposable weigh boat. Move the strip back and forth for 30 seconds.

10.1.2. Check the color on the strip against the color chart on Aquacheck container.

10.1.3. If there is chlorine present, add 80 mg of sodium thiosulfate.

10.1.4. Record procedure on extraction benchsheet.

10.2. pH Determination (aqueous only)

10.2.1. Obtain a pH strip and place it directly into a small amount of sample in a disposable weigh boat. Move the strip back and forth for 30 seconds.

10.2.2. Check the color on the strip against the color chart on the pH container.

10.3. % Solids Determination

10.3.1. "ZERO" or "TARE" the balance.

10.3.2. Place a weigh boat on the balance and record the weight as "Boat Weight".

10.3.3. Samples are individually homogenized with a clean spoon, spoonula or spatula. Add a portion of the sample (2 – 10 g) to the weigh boat and record the weight as "Wet Wt. + Boat Wt."

10.3.4. Place the weigh boat plus sample in an oven at 110±5°C for at least overnight.

10.3.5. Remove the weigh boat plus sample from the oven and allow to come to room temperature.

10.3.6. "ZERO" or "TARE" the balance.

10.3.7. Place the weigh boat plus sample on the balance and record the weight as "Residue + Boat Wt."

10.3.8. Calculate the percent solids by the following formula:

$$\%\text{Solids} = \frac{(\text{Residue Wt.} + \text{Boat Wt.}) - (\text{Boat Wt.})}{(\text{Wet Wt.} + \text{Boat Wt.}) - (\text{Boat Wt.})} \text{ X } 100$$

10.4. Compositing – by client request

10.4.1. Samples are individually homogenized, if necessary, with a clean spoon, spoonula or spatula. Aqueous samples should be mixed and shaken to obtain a representative sample.

10.4.2. Weigh out approximately 50 grams, or amount designated by the client, from each individual sample and place into a pan.

10.4.3. Repeat the homogenization for each sample.

10.4.4. Place each individual sample into a new, separate container. Record the weight of each sample on the benchsheet.

10.4.5. Retain the original sample containers. The new container is given a new sample ID number and then processed through the appropriate extraction.

10.5. Sample Weight Determination (Aqueous)

10.5.1. Volumetric: Allow sample to come to ambient temperature, mark the water meniscus on the side of the 1 L sample bottle. Once the sample has been transferred, fill the sample bottle to the mark with water and transfer to a 1000 mL graduated cylinder. Record the sample volume to the nearest 5 mL.

10.5.2. Gravimetric: Sample bottle including sample is placed on calibrated balance. The weight is recorded. The empty bottle is allowed to air-dry overnight and then re-weighed on a calibrated balance. This weight is recorded.

11. EXTRACTION PROCEDURES

11.1. Aqueous Samples

11.1.1. Record the combined weight of the bottle, cap and sample for each sample to be extracted. After the sample has been removed from the bottle, allow it to drain overnight and reweigh it and the cap to determine the amount of sample extracted.

11.1.2. For the method blank (MB) and OPR(s), transfer ~1 liter of HPLC water into a one liter bottle for each.

11.1.3. Add the appropriate volume of Internal Standard (IS) solution to a test tube containing ~1 mL of acetone.

Quantitatively transfer to the samples and QC with small portions of the solvent used.

11.1.4. Add the appropriate volume of Native Standard (NS) solution to a test tube containing the IS/solvent and then quantitatively transfer to the aliquot of matrix assigned as an LCS, OPR, MS or MSD. Allow the spiked samples to equilibrate for at least 1 hour before extraction.

11.1.5. Pour the sample into a 2-liter separatory funnel. Rinse the sample container with ~60 mL of $MeCl_2$ and add it to the separatory funnel.

11.1.6. Stopper each separatory funnel and shake vigorously, with frequent venting, for 2 minutes.

11.1.7. Allow the phases to separate (centrifugation or other mechanical means may be used to facilitate separation).

11.1.8. Drain the $MeCl_2$ extract through a funnel of Na_2SO_4 into a 500 mL round bottom flask.

11.1.9. Extract the aqueous phase with two more ~60 mL portions of $MeCl_2$ (shaking 1 minute each time) and pass the extracts through the same funnel of Na_2SO_4 into a round bottom flask.

11.1.10. Pass the final extract through Na_2SO_4 one last time and concentrate the extract to approximately 10 mLs.

11.2. Soil Samples

11.2.1. Samples are individually homogenized with a clean spoon, spoonula or spatula. Weigh the sample (nominal 10 g dry weight equivalent) directly into an analyte-free thimble, carefully breaking up any large lumps of sample.

11.2.2. Add the appropriate volume of IS and NS solutions into test tubes containing DCM. Pour into the soxhlet, rinsing three times with DCM.

11.2.3. Assemble the soxhlet apparatus, and add a fresh charge of DCM to the receiver and reflux flask. Apply power to the heating mantle to begin refluxing.

11.2.4. Reflux the sample for a total of 16 hours. Cool and disassemble the apparatus.

11.2.5. Pass extract through Na_2SO_4.

11.2.6. Add CRS and tetradecane to the extract.

11.2.7. Concentrate the extracts using the rotary evaporator. Exchange twice with 50 mLs of hexane. Bring to < 2mL of hexane.

12. CLEANUP PROCEDURES

12.1. Silica Gel Column Preparation (aqueous only)

12.1.1. Pack an 11 mm. id. x 160 mm. column as per Figure 1.

12.1.2. Pre-rinse the column with 20 mL of hexane. Discard the rinsate.

12.1.3. Quantitatively transfer the sample extract onto the column using a disposable pipet. Rinse with additional hexane and add to the column and collect the eluate.

12.1.4. Just prior to the exposure of the sodium sulfate layer to air, add 25 mL of hexane. After the hexane has passed, elute with 35 mL of 40% MeCl2/hexane. Continue collection of the eluate into a 100 mL round bottom flask.

12.1.5. Roto-evaporate the eluate to less than 5 mL. Quantitatively transfer to a conical vial, using a hexane rinse. Concentrate appropriately.

12.2. Acid Base Silica Gel/Acid Alumina (ABSG/AA) (soil/sediments)

12.2.1. Prepare the column as depicted in Figure 2.

12.2.2. Rinse the ABSG column with ~60 mL hexane, discard the eluate. Rinse the AA column with ~30 mL of DCM and then ~30 mL hexane.

12.2.3. Position the ABSG column so that it elutes directly onto the Acid Alumina column.

12.2.4. Transfer the extract to the ABSG column with 2-4 small portions of hexane.

12.2.5. When the extract reaches the sodium sulfate, add 150 mL of hexane.

12.2.6. When all of the ABSG eluate has passed through the Acid Alumina column, remove the ABSG column, discard all the

eluates.

12.2.7. Elute the Acid Alumina column with ~50 mL of 20% MeCl2:hexane, collect the eluate.

12.2.8. Add 100 µL tetradecane and concentrate to the tetradecane at 50°C (only if proceeding to Florisil).

12.3. Florisil (F) (soil/sediments)

12.3.1. Prepare the column as depicted in Figure 3.

12.3.2. After removing the florisil jar from the oven, allow the florisil to cool ~ 10 minutes before weighing out 1 gram.

12.3.3. Rinse the column with ~50 mL of DCM, then ~50 mL of hexane, discard the eluate.

12.3.4. Transfer the extract to the column with 2-4 small portions of hexane, discard the eluate.

12.3.5. Elute the column with ~30 mL of hexane, discard eluate.

12.3.6. Elute the column with ~50 mL of $MeCl_2$, collect the eluate.

12.3.7. Concentrate the eluate appropriately.

13. ADJUST TO FINAL VOLUME

13.1. Using hexane, quantitatively transfer the concentrated eluate to a conical vial that contains the Recovery Standard (RS) and 10 µL of tetradecane.

13.2. Using nitrogen blow down, concentrate to the tetradecane.

13.3. Rinse the walls of the conical with hexane, concentrate down to the tetradecane.

13.4. Using a 10-20 µL Wiretrol, transfer the tetradecane to an insert in a crimp top amber autoinjector vial and then cap.

14. GC/MS ANALYSIS

14.1. Analyze samples with selected ion monitoring.

14.2. Recovery of each internal standard versus the recovery standard must be between 25 – 150% or have a signal to noise ratio > 2.5:1 for natives and > 10:1 for labeled compounds.

15. CALIBRATION

15.1. Initial Calibration

15.1.1. An initial calibration curve is created to demonstrate the linearity of the HRMS system over the calibration range. An initial calibration is repeated whenever a new set of spiking calibration standards are created or whenever the continuing calibration falls outside the acceptance criteria.

15.1.2. Each calibration standard contains HCX. Calibration standard solutions are presented in Table 2.

15.1.3. One internal standard and one recovery standard are used to improve quantitation.

15.1.4. See Table 2 for calibration range.

15.1.5. 2 µL maximum injection of standards are made to create an initial calibration curve whenever the continuing calibration check falls outside the acceptable relative response factor window.

15.1.6. An initial calibration curve is accepted if the following criteria are met:

1.) The signal to noise ratio (s/n) exceeds 10:1 for all ions monitored.

2.) The ion abundance ratio measurements are within ± 15% of the theoretical ratio.

3.) The %RSD for the mean response factors must be within ±20% for the native standards and within ±35% for internal standards.

15.1.7. If the criteria are not achieved, a new initial calibration curve must be prepared and analyzed.

15.2. Continuing Calibration

15.2.1. A verification (VER) standard from the initial calibration curve (CS3) is injected at the beginning of an analytical 12-hour sequence. The following criteria must be met:

15.2.2. The percent deviation recoveries for the native compound must be 70-130% and 50-150% for labeled compounds.

15.2.3. The ion ratios are within the criteria listed in Table 1. If the ratios do not meet the acceptance criteria, then the instrument must be recalibrated and the affected samples are reanalyzed.

15.2.4. The signal to noise ratio (s/n) must exceed 10:1 for all ions monitored. If the s/n ratio is not met, then associated extracts are re-analyzed.

15.3. Qualitative Determination

15.3.1. To identify a chromatographic peak as a HCX, it must meet the following criteria:

1) The signals for the two exact m/zs being monitored must be present and must maximize within ±2 seconds of one another.

2) The signal-to-noise ratio (S/N) of each of the two exact m/zs must be ≥2.5:1 for a sample extract.

3) The ion abundance ratios must be within the limits established for the homologous series (Table 1A and 1B).

4) The relative retention time of HCX must be within 1.044-1.086 seconds of ^{13}C-1,2,3,7,8,9-HxCDF.

15.3.2. Quantitative Determination

15.3.3. Quantitate the HCX peaks from the response relative to the appropriate internal standard.

15.3.4. Recovery of each internal standard versus the recovery standard must be 25-150%.

15.3.5. Recoveries below the limits may be accepted if the signal to noise is >10:1. If the signal to noise is not >10:1, samples must be re-extracted and re-analyzed or the data must be qualified.

15.3.6. If a chromatographic peak saturates the detector, a dilution of the extract must be analyzed.

16. CALCULATIONS

16.1. The concentration for HCX is calculated by using the formula:

$$C_X = \frac{(A_X)(Q_{IS})}{(A_{IS})(W)(RRF)}$$

Where:

C_X	=	*Concentration of unlabeled HCX,*
A_X	=	*Sum of the integrated ion abundances of the quantitation ions for unlabeled HCX*
A_{IS}	=	*Sum of the integrated ion abundances of the quantitation ion for the labeled internal standards,*
Q_{IS}	=	*Quantity, in pg, of the internal standard added to the sample before extraction,*
W	=	*Weight of the sample (solid, dry weight or liquid)*
DW	=	*Sample wt.× %solids/100*
RRF	=	*Calculated relative response factor for the analyte.*

16.2. The detection limits can be calculated using the following formula:

$$DL = \frac{(2.5)(H_N)(Q_{IS})}{(H_{IS})(W)(RRF)}$$

Where:

DL	=	*Sample specific estimated detection limit,*
H_N	=	*Noise height (peak to peak),*
H_{IS}	=	*Peak height of the internal standard,*
Q_{is}	=	*Quantity, in pg, of the internal standard added to the sample before extraction,*
W	=	*Weight of the sample (solid or liquid), and*
RRF	=	*Calculated relative response factor for the analyte.*

16.3. The reporting limits can be calculated using the following formula:

$$RL = \frac{(\text{Extract Conc. of Low point of curve})(\text{Final volume})}{(\text{Weight of sample})}(\text{Split})$$

16.4. The Relative Response factor can be calculated using the following formula:

$$RRF = \frac{\left(A^1{}_N + A^2{}_N\right)(C_{IS})}{\left(A^1{}_{IS} + A^2{}_{IS}\right)(C_N)}$$

Where:

$A1_N$, $A2_N$	=	*Areas of the primary and secondary m/zs for the native compound*
$A1_{IS}$, $A2_{IS}$	=	*Areas of the primary and secondary m/zs for the labeled compound.*
C_{IS}	=	*Concentration of the labeled compound in the calibration standard.*
C_N	=	*Concentration of the native compound in the calibration standard*

17. POLLUTION PREVENTION

17.1. The techniques used in this method are amenable to solvent recovery, and it is recommended that the laboratory recover solvents wherever feasible.

17.2. Standards should be prepared in volumes consistent with laboratory use to minimize disposal of standards.

18. WASTE MANAGEMENT

18.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. Safety officer should be contacted if additional information is required.

18.2. The laboratory waste management is in compliance with all federal, state, and local regulations to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations.

19. REFERENCES

19.1. EPA Method 1668, Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS, United States Office of Water, EPA No. EPA 821-R-00-002, Environmental Protection Agency (4303), December 1999

19.2. USEPA Method 1613, Revision B, Dated October 1994.

Figure 1

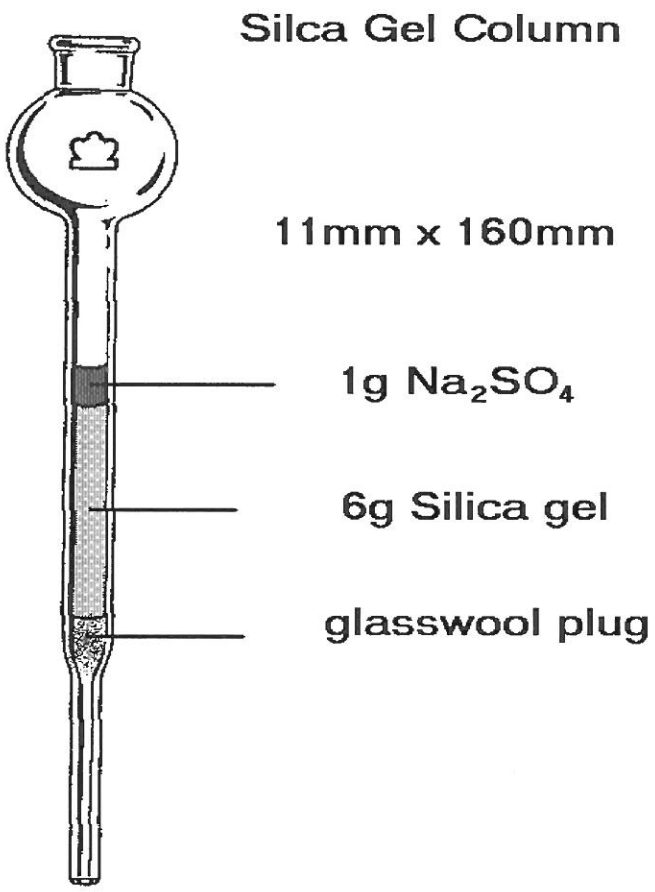

Silca Gel Column
11mm x 160mm
1g Na_2SO_4
6g Silica gel
glasswool plug

Figure 2

Acid Base Silica Gel/Acid Alumina (ABSG/AA)

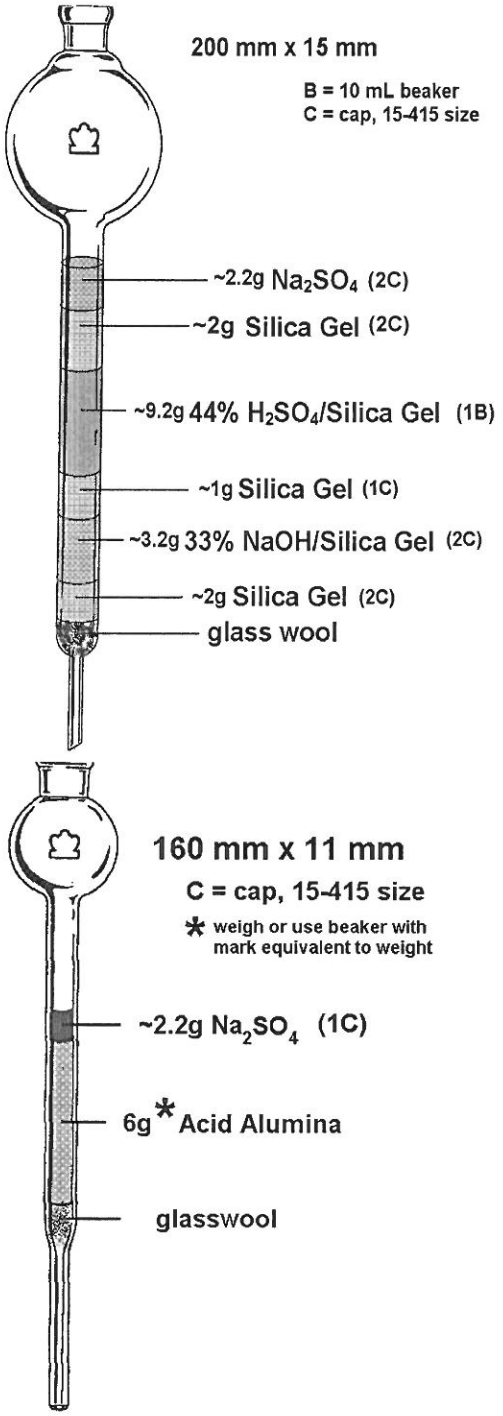


Figure 3

Florisil

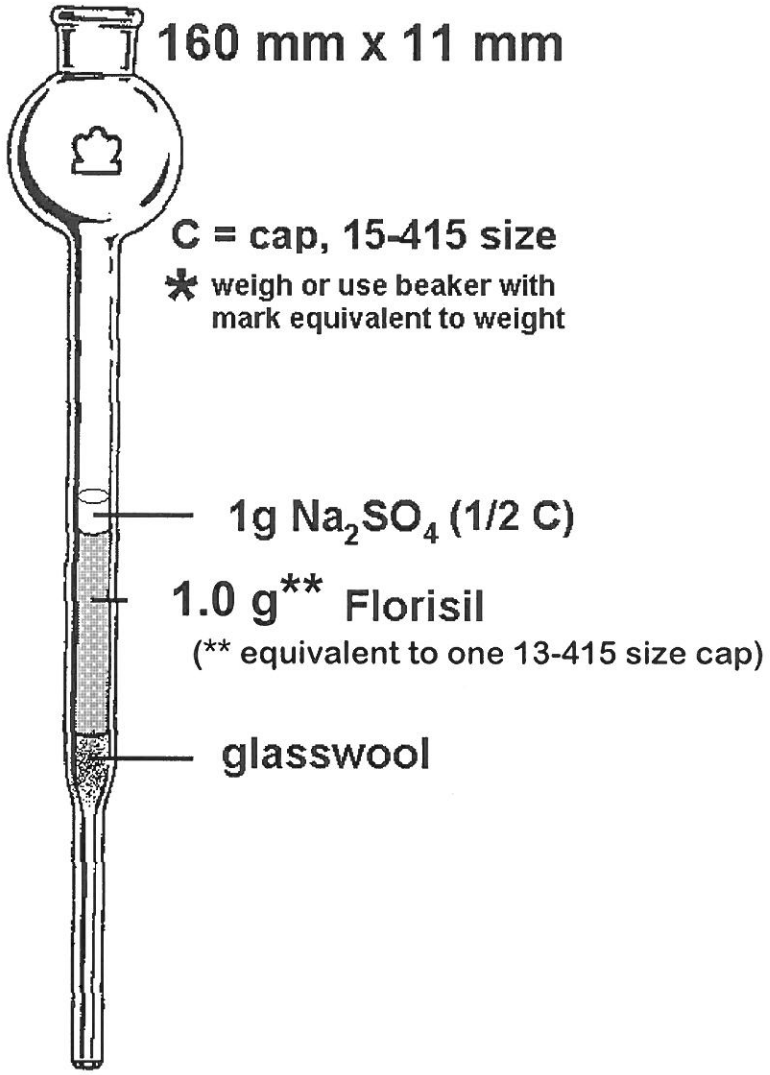


Table 1A

Hexachloroxanthene

Number of Chlorine Atoms	Exact m/z	Theoretical Ion Ratio	Control Limits	
			Lower	Upper
6	387.8364/389.8334	1.24	1.05	1.43

(1) Represents ±15% windows around the theoretical ion abundance ratios.

Table 1B

Standards

Labeled Compound	Exact m/z	Theoretical Ion Ratio	Control Limits	
			Lower	Upper
^{13}C-1,2,3,7,8,9-HxCDF	383.8639/385.8610	0.51	0.43	0.59
^{13}C-1,2,3,4,6,9-HxCDF	383.8639/385.8610	0.51	0.43	0.59
^{13}C-1,2,3,7,8,9-HxCDD	401.8560/403.8530	1.24	1.05	1.43

Table 2

Compound	Calibration Solutions (ng/mL)						
Native Compound	**CS0**	**CS1**	**CS2**	**CS3***	**CS4**	**CS5**	**CS6**
Hexachloroxanthene	10	25	100	500	1000	2000	4000
Labeled Compound							
^{13}C-1,2,3,7,8,9-HxCDF	100	100	100	100	100	100	100
Recovery Standard							
^{13}C-1,2,3,4,6,9-HxCDF	100	100	100	100	100	100	100
Cleanup Recovery Standard							
^{13}C-1,2,3,7,8,9-HxCDD	100	100	100	100	100	100	100

* Calibration Verification Solution

Table 3

Congener	VER	IPR		OPR %	Labeled compound recovery in samples %
		RSD %	Ave %		
1,2,4,5,7,8-HxCX	70-130	40	60-140	50-150	
^{13}C-1,2,3,7,8,9-HxCDF	50-150	50	35-135	30-140	25-150
^{13}C-1,2,3,7,8,9-HxCDD	50-150	50	35-135	30-140	25-150

Glossary

Calibration Standard (CAL) — A solution prepared from a secondary standard and/or stock solutions and used to calibrate the response of the instrument with respect to analyte concentration.

Calibration Verification Standard (VER) — The mid-point calibration standard (CS3) that is used in to verify calibration. See Table 2.

CS0, CS1, CS2, CS3, CS4, CS5, CS6 — See Calibration standards and Table 2.

Field Blank — An aliquot of reagent water or other reference matrix that is placed in a sample container in the laboratory or the field, and treated as a sample in all respects, including exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the field blank is to determine if the field or sample transporting procedures and environments have contaminated the sample.

GC — Gas chromatograph or gas chromatography.

HRGC — High resolution GC.

HRMS — High resolution MS.

IPR — Initial precision and recovery; four aliquots of the diluted PAR standard analyzed to establish the ability to generate acceptable precision and accuracy. An IPR is performed prior to the first time this method is used and any time the method or instrumentation is modified.

Laboratory Blank — See method blank.

Laboratory Control sample (LCS) — See ongoing precision and recovery standard (OPR).

Laboratory Reagent Blank — See method blank.

May — This action, activity, or procedural step is neither required nor prohibited.

May Not — This action, activity, or procedural step is prohibited.

Method Blank — An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with samples. The method blank is used to determine if analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

Minimum Level (ML) — The level at which the entire analytical system must give a recognizable signal and acceptable calibration point for the analyte. It is equivalent to the concentration of the lowest calibration standard, assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed.

MS — Mass spectrometer or mass spectrometry.

Must — This action, activity, or procedural step is required.

OPR — Ongoing precision and recovery sample (OPR); a laboratory blank spiked with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this method for precision and recovery.

PFK — Perfluorokerosene; the mixture of compounds used to calibrate the exact m/z scale in the HRMS.

Preparation Blank — See method blank.

Primary Dilution Standard — A solution containing the specified analytes that is purchased or prepared from stock solutions and diluted as needed to prepare calibration solutions and other solutions.

Quality Control Check Sample (QCS) — A sample containing all or a subset of the analytes at known concentrations. The QCS is obtained from a source external to the laboratory or is prepared from a source of standards different from the source of calibration standards. It is used to check laboratory performance with test materials prepared external to the normal preparation process.

Reagent Water — Water demonstrated to be free from the analytes of interest and potentially interfering substances at the method detection limit for the analyte.

Relative Standard Deviation (RSD) — The standard deviation times 100 divided by the mean. Also termed "coefficient of variation."

RSD — See relative standard deviation.

Should — This action, activity, or procedural step is suggested but not required.

SICP — Selected ion current profile; the line described by the signal at an exact m/z.

Stock Solution — A solution containing an analyte that is prepared using a reference material traceable to EPA, the National Institute of Science and Technology (NIST), or a source that will attest to the purity and authenticity of the reference material.

VER — See calibration verification standard.

Attachment B

EDS Validation SOP
SOP-V1, HCX Rev. 0,
August 2011

Validation SOP
No. V-1

EDS SOP, HCX Rev. 0, 8/11

Environmental Data Services
Validation Guidelines for Hexachloroxanthene

SITE:
DATE:
SDG:
VALIDATOR:

All deliverables must be clearly labeled with the associated sample number. Review the data package to assure that all items listed below are provided. Missing, illegible or incorrectly labeled items must be checked off. The laboratory should immediately be contacted and requested to submit the missing or incorrect items.

	Y	N	N/A
Data Completeness and Deliverables			
Are the Reports and Forms present for all samples?			
Traffic Report	_____	_____	_____
Field Chain of Custody	_____	_____	_____
Laboratory Chain of Custody Records	_____	_____	_____
Sample Shipment Records	_____	_____	_____
% Solid Worksheet	_____	_____	_____
Are the Case Narrative and Cover Letter present?	_____	_____	_____
Do the Field Chain of Custody Reports or Lab Case Narrative indicate problems with sample receipt, sample condition, analytical procedures, or other comments regarding the quality of the data?	_____	_____	_____

ACTION: Use professional judgment to evaluate the effect of the noted problems on the quality of the data.

ACTION: If any solid sample analyzed contains 50% to 90% water, all data shall be flagged as estimated "J". If a solid sample contains more than 90% water, then qualify positive hits "J" and non-detects "R".

Reporting Requirements and Deliverables			
Are the following forms present?			
Sample Data Summary	_____	_____	_____
Initial Calibration Summary	_____	_____	_____
Initial Calibration Ion Ratio Summary	_____	_____	_____
Routine Calibration Summary	_____	_____	_____
A Chronological List of All Sample Analyses	_____	_____	_____
Matrix Spike/Matrix Spike Duplicate Summary	_____	_____	_____
Method Blank Summary	_____	_____	_____

	Y	N	N/A

GC/MS Displays

Are the following GC/MS Displays present?

Standard and sample chromatograms. SICPs list date and time of analysis; the file name; sample number; and instrument I.D. number	_____	_____	_____
Integrated area and peak height must be listed for all peaks 2.5 times above background	_____	_____	_____
SICP for the initial calibration standard	_____	_____	_____
SICP for the continuing calibration standard	_____	_____	_____
SICP for each sample run	_____	_____	_____

Laboratory Records

GC/MS Standard and Sample Run Log in chronological order	_____	_____	_____
Sample Extraction Log	_____	_____	_____

ACTION: If deliverables are missing call the lab for explanation/ resubmittal. If the lab cannot provide missing deliverables, assess the effect on the validity of the data. Note in the reviewers narrative.

Holding Times

Have any holding times been exceeded for:

aqueous samples 14 days from sample collection to extraction	_____	_____	_____
soil/sediment samples 14 days from sample collection to extraction	_____	_____	_____
all samples 40 days from time of extraction to time of analysis	_____	_____	_____

ACTION: If holding times are exceeded, flag all data as estimated "J". If holding times from collection to extraction, or from extraction to analysis have been grossly exceeded, use professional judgment to determine whether non-detects shall be rejected.

Preservation Requirements

Is the cooler temperature ≤ 10°C for aqueous and soil samples from the time of collection until receipt at the laboratory?	_____	_____	_____

ACTION: If cooler temperature >10^{o}C, flag non-detects as "UJ" and detects as "J".

Y N N/A

Instrument Performance

Mass Calibration - Mass calibration of the MS must be performed prior to analyzing calibration solutions, blanks, samples, and QC samples. A static resolving power of at least 10,000 (10% valley definition) must be demonstrated at appropriate masses before any analysis is performed at appropriate masses. Include in the narrative, minimum required resolving power of 10000 was obtained for perfluorokerosene (PFK) ion 380.9760. This is done by first measuring peak width at 5% of the maximum. This should not exceed 100 ppm, i.e., it should not exceed 0.038, for ion 380.9760. Resolving power, then is calculated using the formula,

Resolving Power = m/_m = 380.9760/0.038 = 10025

Was mass calibration performed at the frequency given above? _____ _____ _____

Initial 5-Point Calibration

The initial calibration standard solutions (CS0 - CS6) must be analyzed prior to any sample analysis. However, initial calibration should be analyzed whenever a new set of standards are created and/or continuing calibration falls outside the acceptance criteria. The calibration standards must be analyzed on the same instrument using the same GC/MS conditions that are used to analyze the samples.

The following MS/DS conditions must be used:

Is mass calibration performed as described earlier _____ _____ _____

Is the total scanning time $\leq$ 1 second _____ _____ _____

Were SIM data acquired for each:

Native HCX ions: 387.84 and 389.83 _____ _____ _____

Internal Standard ions: $^{13}C_{12}$-1,2,3,7,8,9-HxCDF 383.86 and 385.86 _____ _____ _____

Recovery Standard ions: $^{13}C_{12}$-1,2,3,4,6,9-HxCDF 383.86 and 385.86 _____ _____ _____

Were the following GC criteria met:

The absolute retention times of internal standard, $^{13}C_{12}$-1,2,3,7,8,9-HxCDF, recovery standard $^{13}C_{12}$-1,2,3,4,6,9-HxCDF and native HCX shall not change by more than 10 seconds between the ICC3 analysis and the analysis of any other standard. _____ _____ _____

	Y	N	N/A
The relative ion abundance criteria for HCX and standards listed in Table 1A and 1B (see analytical method) must be met.	_____	_____	_____
For all calibration solutions, the signal to noise ratio (S/N) for the GC signal present in every SICP, including the ones for the labeled standards must be $\geq$ 10.	_____	_____	_____
The percent relative standard deviations (%RSD) for the mean Response factors (RRF) from the initial calibration for unlabeled standards must not exceed $\pm$ 20%.	_____	_____	_____

ACTION: 1) If the %RSD for any isomer exceeds 20%, flag the associated sample positive results for that specific isomer as estimated ("J"). No effect on the non-detect data.

2) If the ion abundance ratio for an analyte is outside the limits flag the results for that analyte R (reject).

3) If the ion abundance ratio for the internal standard falls outside the QC limits flag the associated positive hits with J. No effect on the non-detects.

4) If the signal to noise ratio (S/N) is below control limits, use professional judgement to determine quality of the data.

5) If the selected monitoring ions specified in Table 1A and 1B were not used for data acquisition, the lab must be asked for an explanation. If an incorrect ion was used, reject all the associated data.

6) If mass calibration criteria as specified earlier is not met, specify that in narrative notes.

7) Non-compliance of all other criteria specified above should be evaluated using professional judgement.

Spot check relative response factor (RRF) calculations and ion ratios. Ensure that the correct quantitation ions for the unlabeled HCX and labeled internal standard and recovery standard were used. In addition verify that the appropriate labeled compound was used.

Y N N/A

To recalculate the RRFs for the Initial Calibration:

For HCX, labeled internal standard compound and recovery standard listed in Table 1A and 1B:

$$RRF_i = \frac{(A_{n1} + A_{n2}) \times Q_{is}}{(A_{is1} + A_{is2}) \times Q_n}$$

Where:

$A_{n1} + A_{n2}$ = The integrated areas of the primary and secondary ions of isomer of interest, HCX

$A_{is1} + A_{is2}$ = The integrated areas of the primary and secondary ions of the appropriate internal standard, $^{13}C_{12}$-1,2,3,7,8,9-HxCDF

Q_{is} = quantity of the appropriate labeled internal standard, $^{13}C_{12}$1,2,3,7,8,9-HxCDF injected (pg)

Q_n = quantity of the unlabeled HCX analyte injected (pg)

Continuing Calibration (HRCC3)

The continuing calibration must be performed at the beginning of a 12 hour period after successful mass resolution and GC resolution performance checks.

Was the continuing calibration run at the required frequency? _____ _____ _____

Was the following MS/DS conditions met:

The total scanning time was $\leq$1 second. _____ _____ _____

ACTION: If the requirement is not met, use professional judgement to determine the validity of the data.

Were SIM data acquired for each:

Native HCX ions: 387.84 and 389.83 _____ _____ _____

Internal Standard ions: $^{13}C_{12}$-1,2,3,7,8,9-HxCDF 383.86 and 385.86 _____ _____ _____

Recovery Standard ions: $^{13}C_{12}$-1,2,3,4,6,9-HxCDF 383.86 and 385.86 _____ _____ _____

	Y	N	N/A
Was following GC criteria must be met:			
The absolute retention time of the internal standards $^{13}C_{12}$1,2,3,4,6,9-HxCDF and $^{13}C_{12}$-1,2,3,7,8,9-HxCDF as well as HCX shall not change by more than 15 seconds between the initial HRCC3 and ending HRCC3 standard analyses.	_____	_____	_____
The %D for the native HCX should fall in the acceptance criteria (70-130%)	_____	_____	_____
The %D for the internal standards should fall in the acceptance criteria (50-150%)	_____	_____	_____
ACTION: When the %D for either native HCX or the internal standards are outside criteria, all data are flagged "J" or "UJ" as appropriate.			
Was the same internal standard compound used to calculate RRF for HCX the initial calibration?	_____	_____	_____
ACTION: If any of these requirements are not met, use professional judgement to determine the validity of the data.			
The two SIM ions for each homolog must maximize simultaneously $\pm$ 2 seconds .	_____	_____	_____
For the CC3 standard solution, the signal to noise ratio (S/N) for the unlabeled HCX ion shall be greater than 10.	_____	_____	_____
For the labeled internal standards, $^{13}C_{12}$1,2,3,7,8,9-HxCDF and the recovery standard $^{13}C_{12}$-1,2,3,4,6,9-HxCDF the signal to noise ratio (S/N) shall be greater than 10.	_____	_____	_____
The relative ion abundance criteria (Table 1A - analytical method) for HCX shall be met.	_____	_____	_____
The relative ion abundance criteria for the internal standard and recovery standard (Table 1B - analytical method) must be met.	_____	_____	_____
ACTION: If any of these requirements are not met reject all data (flag R) directly affected by each specific problem.			
The found concentration for each analyte must fall within the concentration range established by the initial calibration.	_____	_____	_____
ACTION: When the found concentration of the calibration verification sample falls outside the specified allowable range, all data for outlier congeners are flagged J.			
ACTION: If the continuing calibration standard was not analyzed at the required frequency, reject all the data.			

Spot check relative response factor (RRF) calculations and ion ratios. Verify that the appropriate quantitation ions for the HCX, internal standard and recovery standard were used.

Y N N/A

To recalculate RRFs for HCX, and the RRFs for the internal and recovery standard use the following equations:

$$RRF_n = \frac{(A_{n^1} + A_{n^2}) \times Q_{is}}{(A_{is^1} + A_{is^2}) \times Q_n}$$

$$RRF_{is} = \frac{(A_{is^1} + A_{is^2}) \times Q_{rs}}{(A_{rs}^1 + A_{rs^2}) \times Q_{is}}$$

Where:

$A_{n1} + A_{n2}$ = The sum of the areas of the primary and secondary m/z's for the analytes of interest, HCX.

$A_{is1} + A_{is2}$ = The sum of the areas of the primary and secondary m/z's for the internal standard, $^{13}C_{12}$-1,2,3,7,8,9-HxCDF

$A_{rs1} + A_{rs2}$ = The sum of the areas of the primary and secondary m/z's for the recovery standard, $^{13}C_{12}$-1,2,3,4,6,9-HxCDF

Q_{is} = quantity of the appropriate labeled internal standard, $^{13}C_{12}$-1,2,3,7,8,9-HxCDF, injected (pg)

Q_n = quantity of the unlabeled HCX analyte injected (pg)

Q_{rs} = quantity of the labeled recovery standard, $^{13}C_{12}$-1,2,3,4,6,9-HxCDF

RRF_n = Relative response factor from the continuing calibration

To calculate percent difference use the following equation:

$$\% \text{ Difference} = \frac{(RRFi - RRF_n) \times 100}{RRFi}$$

Where:

RRF_i = Relative response factor established during initial calibration.

RRF_n = Relative response factor established during continuing calibration.

Sample Data

The following MS/DS conditions were used:

Scanning time was $\leq$1 second. _____ _____ _____

SIM data were acquired for each of the ions listed in Table 1A and 1B (see analytical method) _____ _____ _____

Identification Criteria

		Y	N	N/A
B.	The absolute retention time for HCX must be within +/-2 percent of the labeled internal standard, $^{13}C_{12}$-1,2,3,7,8,9-HxCDF.	_____	_____	_____
	ACTION: Reject (R) all positive data for the HCX compound.			
	The integrated ion current for each characteristic ion of the HCX analyte must be at least 2.5 times background noise and must have not saturated the detector.	_____	_____	_____
	ACTION: If the integrated ion HCX criteria is not at least 2.5 times background and the detector is saturated but all other criteria are met, qualify all positive data of the specific analyte with J.			
	The integrated ion current for the labeled internal standard compounds characteristic ions must be at least 10 times background noise.	_____	_____	_____
	ACTION: If the integrated ion current for the internal standard ion characteristics are not met but all other requirements are met qualify the positive data of the corresponding analytes with "J".			
	The relative ion abundance criteria (Table 1A - analytical method) for all HCX found present must be met.	_____	_____	_____
	ACTION: If the HCX reported positive do not meet ion abundance criteria, reject (R) all positive data for this analyte. Change the positive values to EMPC (estimated maximum possible concentration).			
	The relative ion abundance criteria for the labeled internal standard and recovery standard must be met (Table 1B - analytical method).	_____	_____	_____
	ACTION: If labeled internal standards $^{13}C_{12}$-1,2,3,7,8,9-HxCDF and recovery standard $^{13}C_{12}$-1,2,3,4,6,9-HxCDF do not meet ion abundance criteria (Table 1B - analytical method) but they meet all other criteria flag all corresponding data with "J".			
	The identification of a GC peak as a PCDF can only be made if no signal having a S/N $\geq$ 2.5 is detected at the same time in the corresponding polychlorinated diphenyl ether channel. Is the above condition met?	_____	_____	_____
	The HCX concentration must be within the calibration range. If not, dilution should have been made to bring the concentration within the calibration range. Was the above criteria met?	_____	_____	_____
	ACTION: Flag HCX results that are not within the calibration range “J” estimated.			
	Do any lock mass ion signals show peak deflections of greater than 50% in the retention time areas of any analyte?	_____	_____	_____

Y N N/A

ACTION: If any peak deflection is greater than 50%, then qualify the associated compound with a "J".

Spot check calculations for positive data and verify that the same labeled internal standards used to calculate RRFs were used to calculate concentration and EMPC.

To recalculate the concentration of individual HCX isomers in the sample use the following equation:

ALL MATRICES OTHER THAN WATER

$$Cn\ (pg/g) = \frac{(A_{n1} + A_{n2}) \times Q_{is}}{W \times (A_{is1} + A_{is2}) \times RRF_n}$$

WATER

$$Cn\ (pg/L) = \frac{(A_{n1} + A_{n2}) \times Q_1}{V \times (A_{is1} + A_{is2}) \times RRF_n}$$

Where:

A_{n^1} and A_{n^2} = The sum of the areas of the primary and secondary m/z's for the analye of interest, HCX.

A_{is^1} and A_{is^2} = The sum of the areas of the primary and secondary m/z's for the Internal standard, $^{13}C_{12}$-1,2,3,7,8,9-HxCDF

Cn = concentration of analyte (pg/g, pg/L)
W = Weight (g) of sample extracted
V = Volume (ml) of sample extracted
Q_{is} = Quantity (pg) of the appropriate labeled internal standard ($^{13}C_{12}$-1,2,3,7,8,9-HxCDF) compound added to the sample prior to extraction.
RRF_n = Calculated relative response factor from continuing calibration for the analyte HCX.

Estimated Detection Limits (EDL)

Was an EDL calculated for each HCX that was not identified _____ _____ _____

Use the equation below to check EDL calculations:

ALL MATRICES OTHER THAN WATER

$$EDL\ (pg/g) = \frac{2.5 \times Q_{is} \times H_{x^1} \times D}{W \times H_{is^1} \times RRFn}$$

WATER

$$EDL\ (pg/L) = \frac{2.5 \times Q_{is} \times H_{x^1} \times D}{V \times H_{is^1} \times RRFn}$$

Y N N/A

Where:

H_{x^1} = peak height of the noise for the ion of the analyte of interest, HCX

V = Volume of the sample (ml)

W = Weight of the sample (g)

H_{is^1} = peak height of the quantitation ion of the internal standard, $^{13}C_{12}$-1,2,3,7,8,9-HxCDF

RRFn = Calculated relative response factor for the analyte

D = dilution factor

Q_{is} = Quantity (pg) of the internal standard added to the sample before extraction.

NOTE: The validator should check the EDL data to verify that peak heights and not areas were used for this calculation. If the area algorithm was used, the validator should contact the laboratory for recalculation.

Estimated Maximum Possible Concentration (EMPC)

Was an EMPC calculated for HCX that had S/N ratio for the quantitation and confirmation ions greater than 2.5, but did not meet all the identification criteria? _____ _____ _____

Use the equation below to check the EMPC calculations:

ALL MATRICES OTHER THAN WATER

$$EMPC\ (pg/g) = \frac{(A_{n1} + A_{n2}) \times Q_{is} \times D}{W \times (A_{is1} + A_{is2}) \times RRFn}$$

WATER

$$EMPC\ (pg/L) = \frac{(A_{n1} + A_{n2}) \times Q_{is} \times D}{V \times (A_{is1} + A_{is2}) \times RRFn}$$

NOTE: See previous sections for definitions

ACTION: If the spot check calculations yielded EDLs or EMPCs different from those reported in Form I, contact the laboratory for an explanation.

	Y	N	N/A

Method Blanks

Has a method blank per matrix been extracted and analyzed with each batch of 20 samples _____ _____ _____

If samples of same matrix were analyzed in different events (i.e. different shifts or days) has one blank for each matrix been extracted and analyzed for each event _____ _____ _____

ACTION: If the proper number of method blanks were not analyzed, notify the contractor. If they are unavailable, reject all positive sample data. However, the reviewer may also use professional judgement to accept or reject positive sample data if no blank was run.

Native Hexachloroxanthene levels measured in the method blank must be less than the quantitation limit or five times lower than the concentration found in any sample within the analytical batch.

Was HCX found at a concentration greater than the PQL in the associated method blank _____ _____ _____

If yes, has the method blank and associated samples been re-extracted _____ _____ _____

ACTION: If no, prepare a list of samples associated with each of the contaminated method blank(s) which have not been re-extracted

ACTION: If the concentration of HCX in the sample is < than 5 times the concentration in the blank, qualify the data as a non-detect “U”.

ACTION: If the concentration of the HCX in the sample is > than 5 times the concentration in the blank, no action is needed.

Rinsate Blank

One rinsate blank must be collected for each batch of 20 soil samples or one per day whichever is more frequent. _____ _____ _____

Do any rinsate blanks show the presence of HCX that is >PQL _____ _____ _____

ACTION: If any rinsate blank was found to be contaminated with any of the HCX document in case narratives.

	Y	N	N/A

Ongoing Precision and Recovery (OPR)

The laboratory must analyze an OPR after the analysis of the calibration verification and before the analysis of any sample in each set.

Was the OPR standard analyzed at the required frequency _____ _____ _____

Did the OPR standard pass the concentration criteria limits:

HCX - (50-150%) _____ _____ _____

^{13}C-1,2,3,7,8,9-HxCDF (30-140%) _____ _____ _____

NOTE: If the OPR of an isomer is outside the recommended control limits and the internal standard recovery of that isomer in the associated samples is also out of range, than the sample and the OPR should be re-extracted and analyzed.

ACTION: If the criteria has not been met, contact the laboratory. If a decision is made to continue with validation, all data at a minimum should be qualified as estimated "J".

Initial Precision and Recovery (IPR)

Was an initial precision and accuracy demonstration performed for the appropriate matrix as per method _____ _____ _____

Were the results of the IPR evaluation acceptance when compared to the acceptance limits in Method1668A _____ _____ _____

ACTION: If IPR date was not provided by the laboratory, contact the lab to obtain the results of the IPR study.

ACTION: If the results of the laboratories IPR study do not meet the acceptance criteria for performance tests based on criteria in Method 1668A contact the lab to initiate remediation of technical difficulties.

Internal Standard Recovery

Were the samples spiked with the labeled internal standard, $^{13}C_{12}$-1,2,3,7,8,9-HxCDF compound as specified in the method? _____ _____ _____

Was the labeled internal standard compound recovery within the required (25-150%) limits? _____ _____ _____

	Y	N	N/A
If not, were samples reanalyzed?	_____	_____	_____

ACTION: If the labeled internal standard recovery was below 25 percent, reject (R) all associated non-detect data (EMPC/EDL) and flag with "J" all positive data.

ACTION: If the labeled internal standard recovery is above the upper limit (150 percent) flag all associated data (positive and non-detect data) with "J".

ACTION: If the labeled internal standard recovery is less than 10% qualify all associated data R (Reject).

Calculate the percent recovery of labeled internal standard compound (Ris) in the sample extract using the following equation.

$$\%\ \mathrm{Recovery} = \frac{(A_{is1} + A_{is2})(C_{rs})}{(A_{rs1} + A_{rs2})(C_{is})(RRF)}\ x\ 100$$

Where:

$A_{is1} + A_{is2}$	=	The integrated areas of the primary and secondary m/z's for the internal standard $^{13}C_{12}$-1,2,3,7,8,9-HxCDF
$A_{rs1} + A_{rs2}$	=	The integrated areas of the primary and secondary m/z's for the internal standard $^{13}C_{12}$-1,2,3,4,6,9-HxCDF
C_{is}	=	The concentration of the internal standard
C_{rs}	=	The concentration of the recovery standard
RRF_n	=	Calculated Relative Response Factor for the analyte.

	Y	N	N/A

Internal Standards

There are no contractual criteria for the Internal Standard area. However, because it is very critical in determining instrument sensitivity, the internal standard area must be checked for every sample.

Are the internal standard areas for every sample and blank within the upper and lower limits of each associated ICC3? _____ _____ _____

Area upper limit = +100% of recovery standard area.
Area lower limit = - 50% of recovery standard area.

Is the retention time of each internal standard within 10 seconds of the associated ICC3? _____ _____ _____

ACTION: If the internal standard area is outside the upper or lower limits flag all related positive and non-detect data (EMPC/EDL) with "J" regardless whether the labeled analog compound recoveries met specifications or not.

If extremely low area counts (<25%) are reported flag all associated non-detect data as unusable (R) and the positive data J.

If the retention time of the internal standard differs by more than 10 seconds from the ICC3 use professional judgement to determine the effect on the results. A time shift of more than 10 seconds may cause certain analytes to elute outside the retention time window by the GC column performance check solution.

Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Sample ID of the sample chosen for MS/MSD analysis: _____

For every sample delivery group of 20 or fewer environmental samples was one MS/MSD pair analyzed _____ _____ _____

Is the MS recovery within the 50 to 150% acceptance range _____ _____ _____

ACTION: If the recovery is out of the 50-150% range and the sample concentration is less than four times the spike concentration, qualify as estimated "J", the analyte in the sample used for MS/MSD.

Does the precision of the MS/MSD analyses meet the $\leq$ 20% RPD criteria _____ _____ _____

ACTION: Qualify the value for that analyte in the sample used for the MS/MSD as estimated "J"; use professional judgment in association with signal to noise ratios and internal standard recoveries for the associated sample data to determine the effect on the quality of the associated sample data.

	Y	N	N/A
Field Duplicate Subsamples			
Sample IDs of the field duplicate pair: _____			
For every batch of 20 samples or less collected there must be a sample designated as duplicate.	_____	_____	_____
For Soil: RPD ≤ 50% when target is detected in both field duplicate samples at ≥ 5x PQL, or concentration differs by less than 2x the PQL when detects are < 5x PQL for both field duplicate samples.			

Data Validation Qualifiers

Qualifier	Description
J	Estimated value (bias undetermined) – The analyte was positively identified; but the associated numerical value is the approximate concentration of the analyte in the sample.
UJ	Estimated non-detect - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
M	The analytical result reported was obtained from a sediment sample found to contain between 50 and 90 percent moisture and had no other data qualifiers added during the data validating process.
EMPC	Estimated Maximum Possible Concentration (EMPC).
R	The sample results are rejected. Due to a significant QA/QC problem, the analysis is invalid and provides no information as to whether the analyte is present or not.

Attachment C

IPR Study Solid (Full Laboratory Deliverable)

IPR
Hexachloroxanthene

Vista Analytical Laboratory

Hexachloroxanthene IPR

Analyte	Spike (pg/g)	IPR1	IPR2	IPR3	IPR4	Ave	Method Limits	%RSD	Method Criteria
1,2,4,5,7,8-HCX	400.0	375.0	412.0	411.0	386.0	396.0	240-560	4.7	40.0
^{13}C-1,2,3,7,8,9-HxCDF	200.0	198.0	187.0	194.0	191.0	192.5	70-270	2.4	50.0
^{13}C-1,2,3,7,8,9-HxCDD	200.0	200.0	192.0	197.0	197.0	196.5	70-270	1.7	50.0
^{13}C-1,2,3,4,6,9-HxCDF	200.0	200.0	200	200.0	200.0	200.0	70-270	0.0	50.0

1,2,4,5,7,8-HxCX	**%Rec**	93.8	103.0	102.8	96.5	99.0	60-140	4.7	40.0
^{13}C-1,2,3,7,8,9-HxCDF	**%Rec**	99.0	93.5	97.0	95.5	96.3	35-135	2.4	50.0
^{13}C-1,2,3,7,8,9-HxCDD	**%Rec**	100.0	96.0	98.5	98.5	98.3	35-135	1.7	50.0

Matrix: Soil
Chemist: CV
7/20/2011
Batch: 3903
using IPR limits from 1668A

PROCESS SHEET

Project No.-AR: 27592-254 of 256

Prep Due: 7/20/2011
Hold Due: 6/7/2012
Client: Vista Analytical Laboratory(AALCA01D)
Client Manager: Martha M. Maier
Method: Hexachlorophene | Hexachlorophene
Hexachloroxanthene | Hexachloroxanthene

Project Due: 7/14/2011
TAT: 21

Split Type: 3903

Matrix: Soil

LabID	Recon	Client-ID	Description	Date Received	SLoc	Shelf
047	☐	IPR-Soil-1		6/8/2011		
048	☐	IPR-Soil-2		6/8/2011		
049	☐	IPR-Soil-3		6/8/2011		
050	☐	IPR-Soil-4		6/8/2011		

Instructions:
cover soxhlets ; silica gel column (like PAHs)

Report Options

Report Level:
EDD Type:
Report Group: Dioxins NoMDL with %Solid

TEQ Type:
Samples Reconciled By: _____ / /

Vial Box ID: Lowa

Date Requested 7/3/2011

Extraction Set: 3903

Project: 27592 **Chemist:** C. Vredevoe 7/20/11

Method(s): Hexachlorophene/Hexachloroxanthene | Hexachlorophene/Hexachloroxanthene **Prep time:** 1332

C	VISTA Sample ID	G Eqv	Sample Amt. (g)	IS/NS CHEM/ WIT DATE	CRS CHEM/WIT DATE	AP CHEM/Date	~~ABSG~~ SG CHEM/Date	AA CHEM/Date	Florisil CHEM/Date	RS CHEM/WIT DATE
☐	0_3903_MB001	N/A	(10.00)	CV N/A 7/20/11	CV FEB 7/21/11	N/A	CV 7/21/11	N/A	N/A	CV FEB 7/21/11
☐	27592_3903_047	↓	↓	↓	↓	↓	↓	↓	↓	↓
☐	27592_3903_048	↓	↓	↓	↓	↓	↓	↓	↓	↓
☐	27592_3903_049	↓	↓	↓	↓	↓	↓	↓	↓	↓
☐	27592_3903_050	↓	↓	↓	↓	↓	↓	↓	↓	↓

IS Name	NS Name	CRS Name	RS Name
PCDD/F	PCDD/F	PCDD/F	PCDD/F
PCB	PCB	PCB	PCB
HCX 110706A 10µL	HCX 110701A 10µL	HCX 110503B 10µL	HCX 110503C 10µL

Cycle Time 7/20 Start: 1450 Stop: 0720 7/21

APP.: SEFUN (SOX) SDS

SOLV: DCM

Other: N/A

Final Volume(s): 20µL CH

Check Out: Chemist: N/A / /

Check-In: Chemist: / /

Balance ID:

Comments:

hrmsgenprepL.rpt

CALIBRATION STANDARDS REVIEW CHECKLIST

Vista Analytical Laboratory

Beg. Calibration ID: ST110721D2-1

End Calibration ID: NA

	Beg.	End
Ion abundance within QC limits?	✓	NA
Concentration within range?	✓	
First and last eluters present?	✓	
Retention Times within criteria?	✓	
Verification Std. named correctly? (ST-Year-Month-Day-VG ID)	✓	
Forms signed and dated?	✓	
Correct ICAL referenced?	✓	
Run Log:		
-Data file matches Conc Cal ID?	✓	
-Correct instrument listed?	✓	
-Samples within 12-hour clock?	y	n

	Beg.	End
Mass resolution > 10,000? ▪ Method 1614 > 5,000; CARB 429 > 8,000	✓	✓
TCDD/TCDF valleys < 25%?	NA	NA
Peaks integrated correctly?	✓	
Manual integrations included?	✓	
8280 CS1 Ending Standard		
-Ratios within limits		
-S/N > 2.5:1		
-CS1 within 12-hour clock		Y

Comments:

Reviewed by: FEB 7/22/11
Initials & Date

* *Ending standard criteria applicable to 8290 only.*

Vista Analytical Laboratory - Injection Log Run file: 110721D2 Instrument ID: VG-7 GC Column ID: db-5

Data file	S#	Sample ID	Analyst	Acq date	Acq time	CCal	ECal
110721D2	1	ST110721D2-1	MAS	22-JUL-11	02:45:14	ST110721D2-1	NA
110721D2	2	SOLVENT BLANK	MAS	22-JUL-11	03:27:45	ST110721D2-1	NA
110721D2	3	0_3903_MB001	MAS	22-JUL-11	04:10:39	ST110721D2-1	NA
110721D2	4	27592_3903_047	MAS	22-JUL-11	04:53:06	ST110721D2-1	NA
110721D2	5	27592_3903_048	MAS	22-JUL-11	05:35:35	ST110721D2-1	NA
110721D2	6	27592_3903_049	MAS	22-JUL-11	06:18:08	ST110721D2-1	NA
110721D2	7	27592_3903_050	MAS	22-JUL-11	07:00:40	ST110721D2-1	NA
110721D2	8	SOLVENT BLANK	MAS	22-JUL-11	07:43:11	ST110721D2-1	NA
110721D2	9	ST110721D2-2	MAS	22-JUL-11	08:25:42	ST110721D2-1	NA

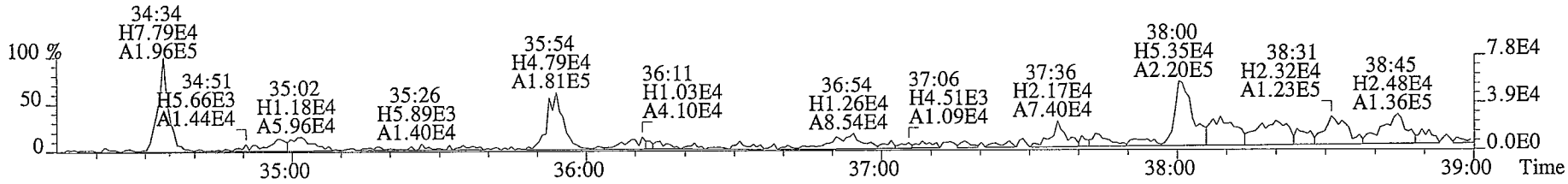
Client ID: 0_3903_MB001 Filename: 110721D2 S:3 Acq:22-JUL-11 04:10:39 ConCal:
Lab ID: 0_3903_MB001 GC Column ID: db-5 ICal: HCXVG7-7-19-11 wt/vol:10.000 EndCAL:

	Name	Resp	RA		RRF	RT	Conc	Qualifiers	Rec	noise	Fac	DL
Unk	1,2,4,5,7,8-HCX		*	n	0.14	NotF	*			8970	2.5	3.95
IS	13C-1,2,3,7,8,9-HxCDF	4.44e+07	0.51	y	0.91	35:54	193		96.5			
C/Up	13C-1,2,3,7,8,9-HxCDD	3.51e+07	1.29	y	0.71	35:29	197		98.4			
RS/R	13C-1,2,3,4,6,9-HxCDF	5.03e+07	0.52	y	1.00	34:34	200					

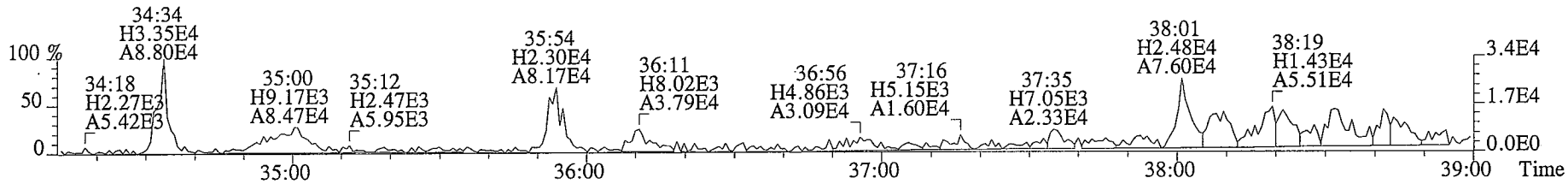
Analyst: MJ

Date: 7/22/11

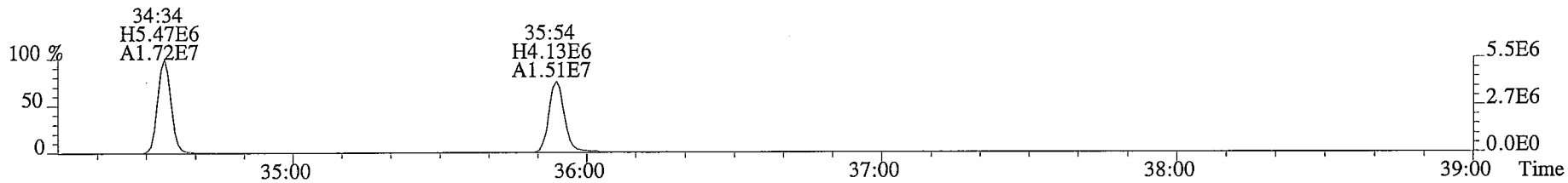
File:110721D2 #1-2305 Acq:22-JUL-2011 04:10:39 GC EI+ Voltage SIR Autospec-UltimaE
Sample#3 File Text:Vista Analytical Laboratory VG-7 Text:0_3903_MB001 Exp:HCX_DB5
387.8364 S:3 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



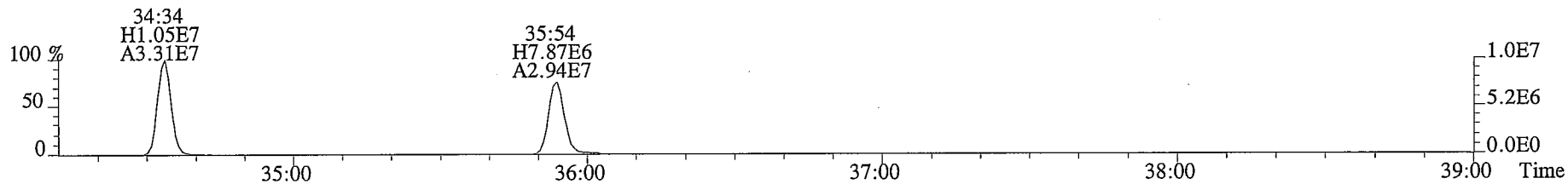
389.8334 S:3 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



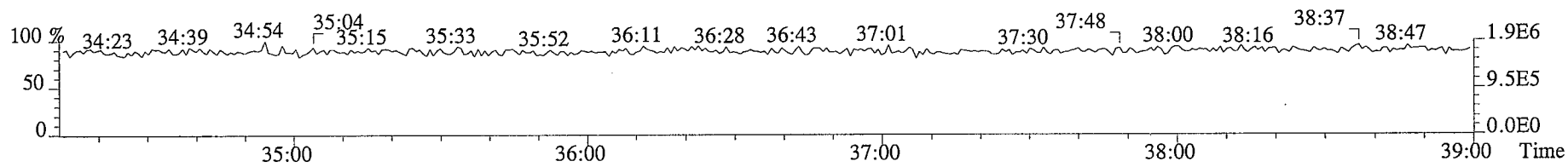
383.8639 S:3 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



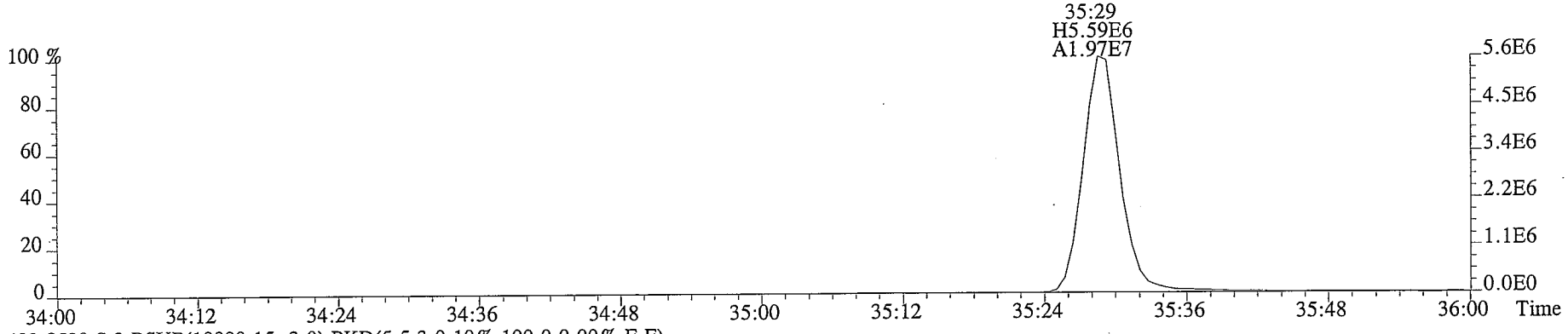
385.8610 S:3 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



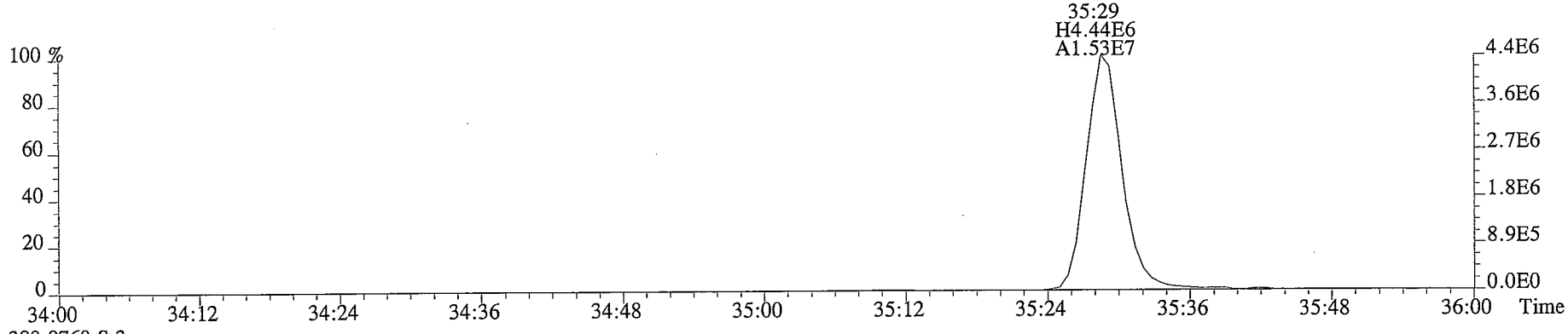
380.9760 S:3



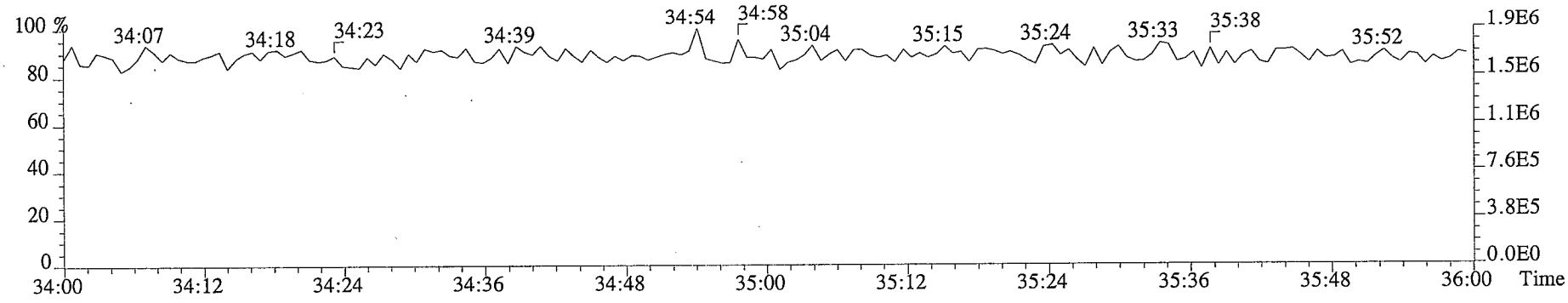
File:110721D2 #1-2305 Acq:22-JUL-2011 04:10:39 GC EI+ Voltage SIR Autospec-UltimaE
Sample#3 File Text:Vista Analytical Laboratory VG-7 Text:0_3903_MB001 Exp:HCX_DB5
401.8560 S:3 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



403.8530 S:3 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



380.9760 S:3



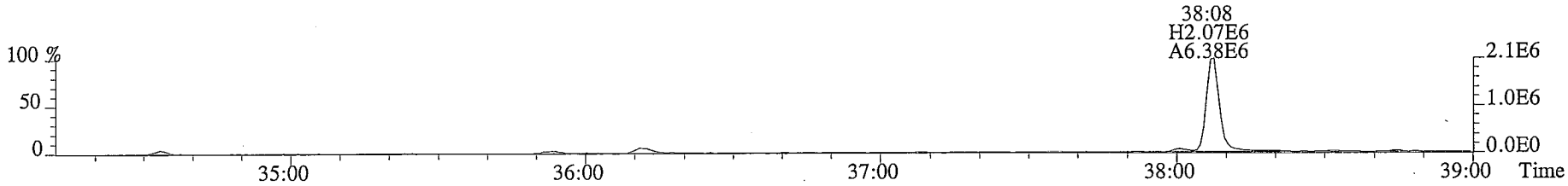
Client ID: IPR-Soil-1 10g Filename: 110721D2 S:4 Acq:22-JUL-11 04:53:06 ConCal:
Lab ID: 27592_3903_047 GC Column ID: db-5 ICal: HCXVG7-7-19-11 wt/vol:10.000 EndCAL:

	Name	Resp	RA	RRF	RT	Conc	Qualifiers	Rec	noise	Fac	DL
Unk	1,2,4,5,7,8-HCX	1.13e+07	1.30 y	0.14	38:08	375			*	2.5	*
IS	13C-1,2,3,7,8,9-HxCDF	4.22e+07	0.53 y	0.91	35:53	198		98.9			
C/Up	13C-1,2,3,7,8,9-HxCDD	3.30e+07	1.29 y	0.71	35:29	200		99.8			
RS/R	13C-1,2,3,4,6,9-HxCDF	4.67e+07	0.53 y	1.00	34:33	200					

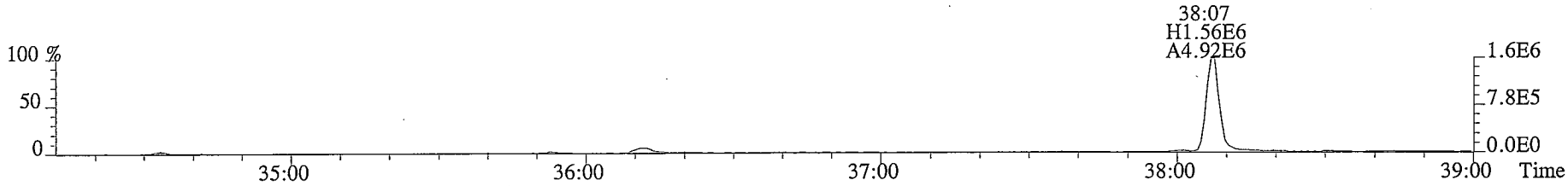
Analyst: VM

Date: 7/22/11

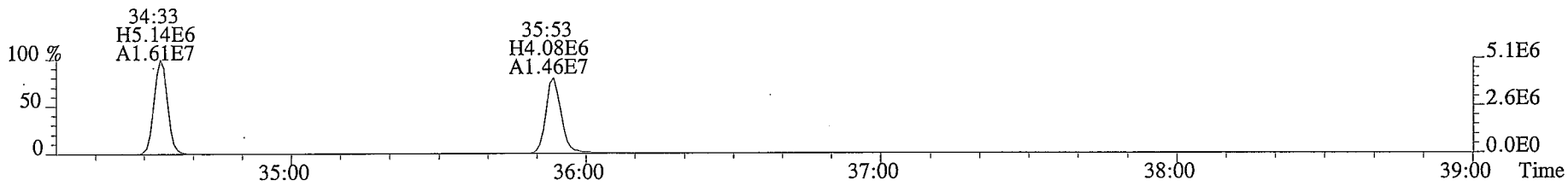
File:110721D2 #1-2306 Acq:22-JUL-2011 04:53:06 GC EI+ Voltage SIR Autospec-UltimaE
Sample#4 File Text:Vista Analytical Laboratory VG-7 Text:27592_3903_047 IPR-Soil-1 10g Exp:HCX_DB5
387.8364 S:4 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



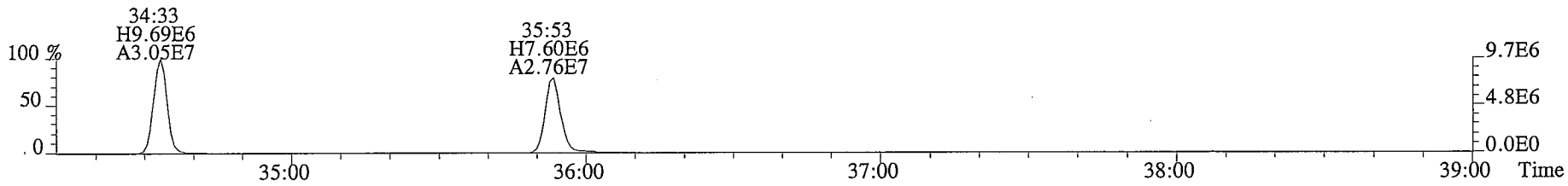
389.8334 S:4 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



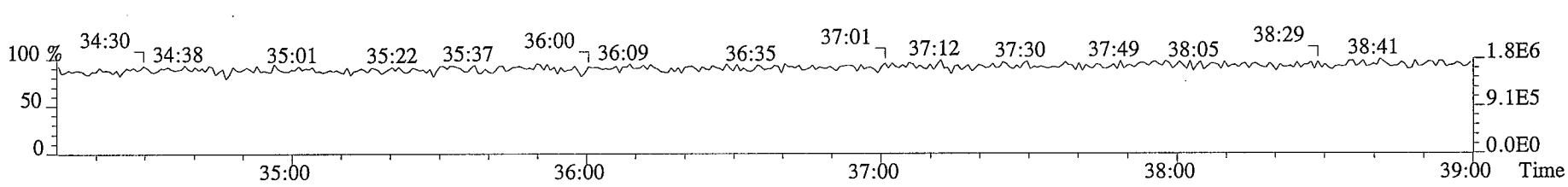
383.8639 S:4 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



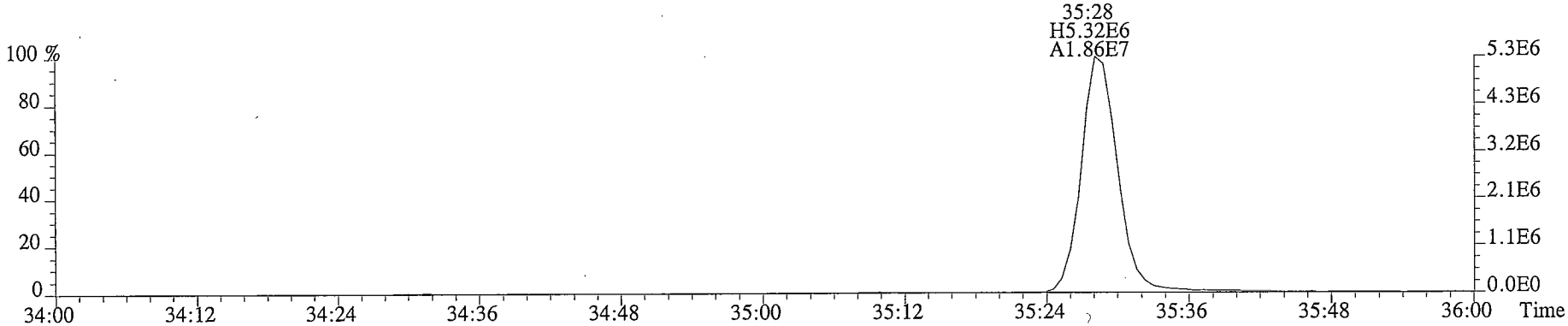
385.8610 S:4 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



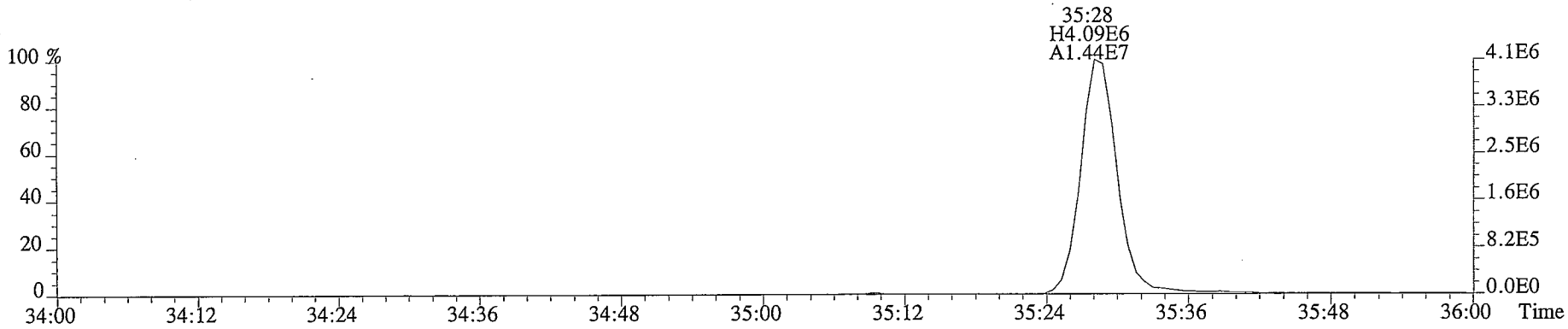
380.9760 S:4



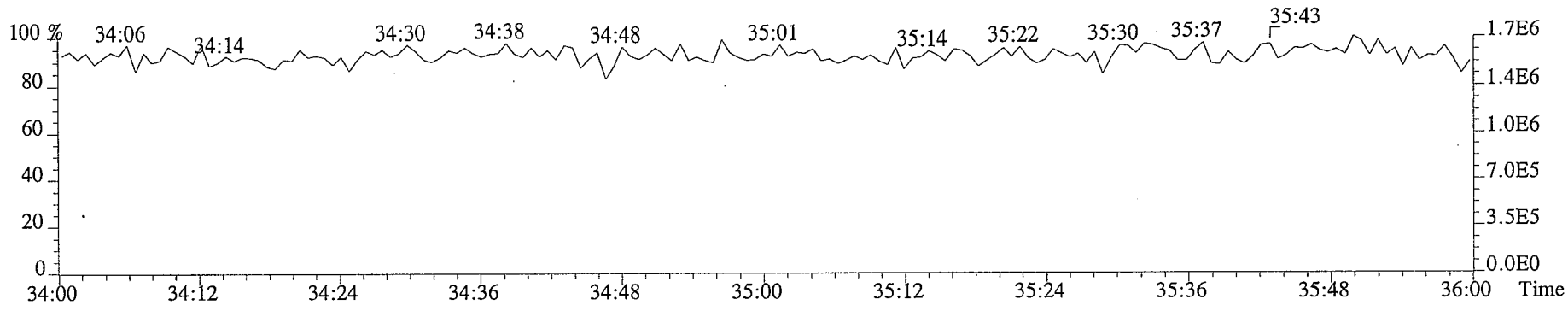
File:110721D2 #1-2306 Acq:22-JUL-2011 04:53:06 GC EI+ Voltage SIR Autospec-UltimaE
Sample#4 File Text:Vista Analytical Laboratory VG-7 Text:27592_3903_047 IPR-Soil-1 10g Exp:HCX_DB5
401.8560 S:4 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



403.8530 S:4 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



380.9760 S:4



Client ID: IPR-Soil-2 10g
Lab ID: 27592_3903_048

Filename: 110721D2 S:5 Acq:22-JUL-11 05:35:35
GC Column ID: db-5 ICal: HCXVG7-7-19-11 wt/vol:10.000

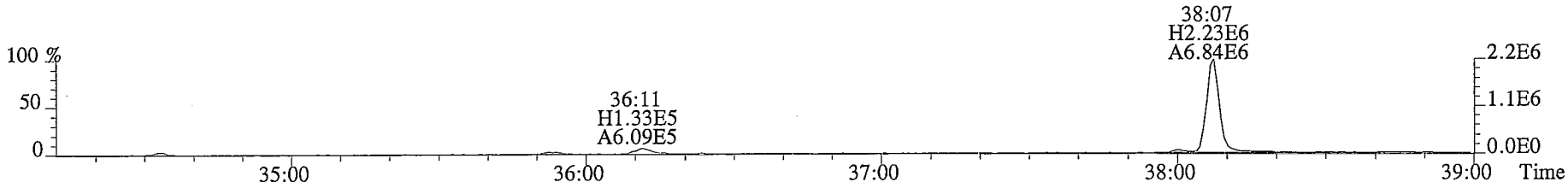
ConCal:
EndCAL:

	Name	Resp	RA	RRF	RT	Conc	Qualifiers	Rec	noise	Fac	DL
Unk	1,2,4,5,7,8-HCX	1.21e+07	1.30 y	0.14	38:07	412			*	2.5	*
IS	13C-1,2,3,7,8,9-HxCDF	4.11e+07	0.53 y	0.91	35:54	187		93.5			
C/Up	13C-1,2,3,7,8,9-HxCDD	3.27e+07	1.28 y	0.71	35:28	192		95.9			
RS/R	13C-1,2,3,4,6,9-HxCDF	4.81e+07	0.53 y	1.00	34:33	200					

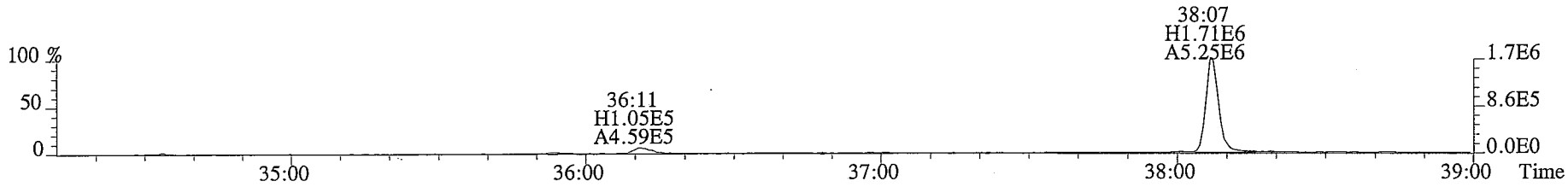
Analyst: VM

Date: 7/22/11

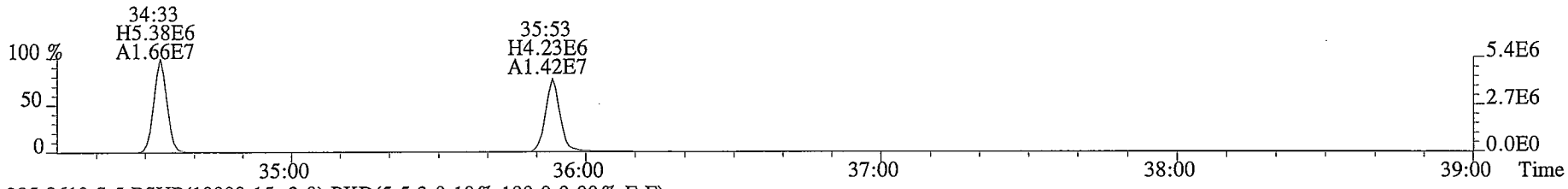
File:110721D2 #1-2305 Acq:22-JUL-2011 05:35:35 GC EI+ Voltage SIR Autospec-UltimaE
Sample#5 File Text:Vista Analytical Laboratory VG-7 Text:27592_3903_048 IPR-Soil-2 10g Exp:HCX_DB5
387.8364 S:5 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



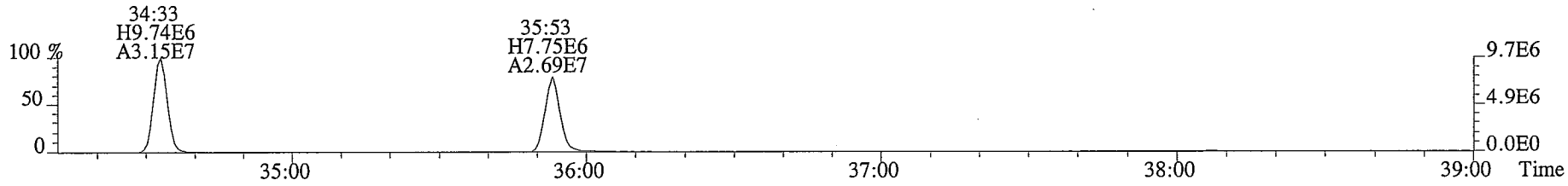
389.8334 S:5 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



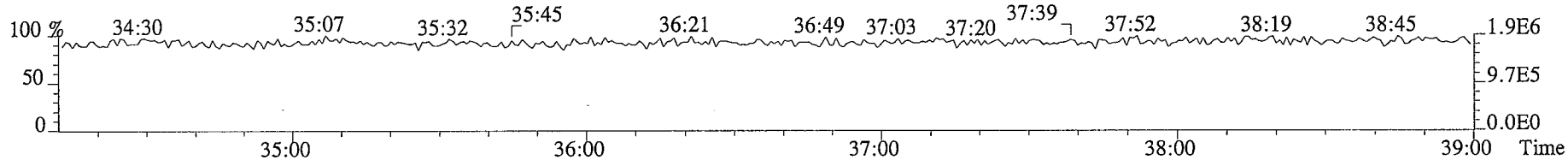
383.8639 S:5 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



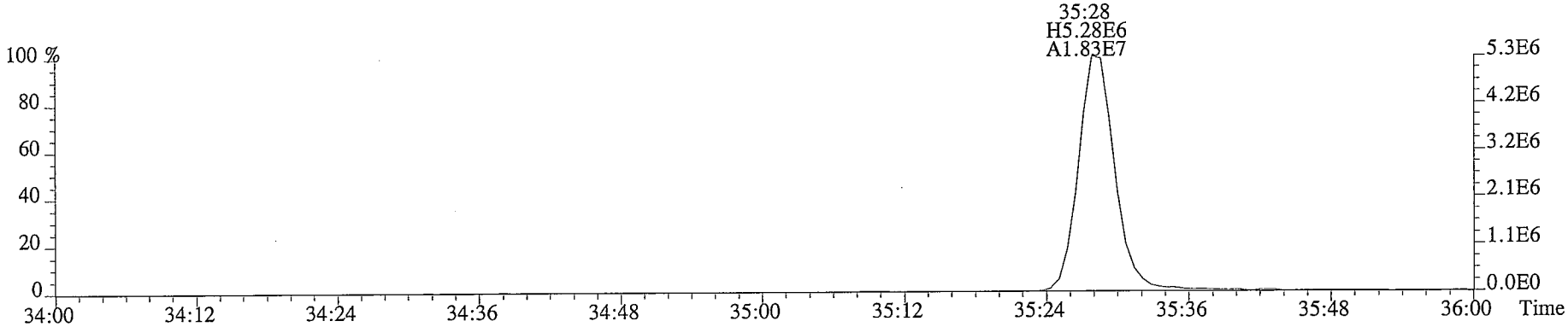
385.8610 S:5 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



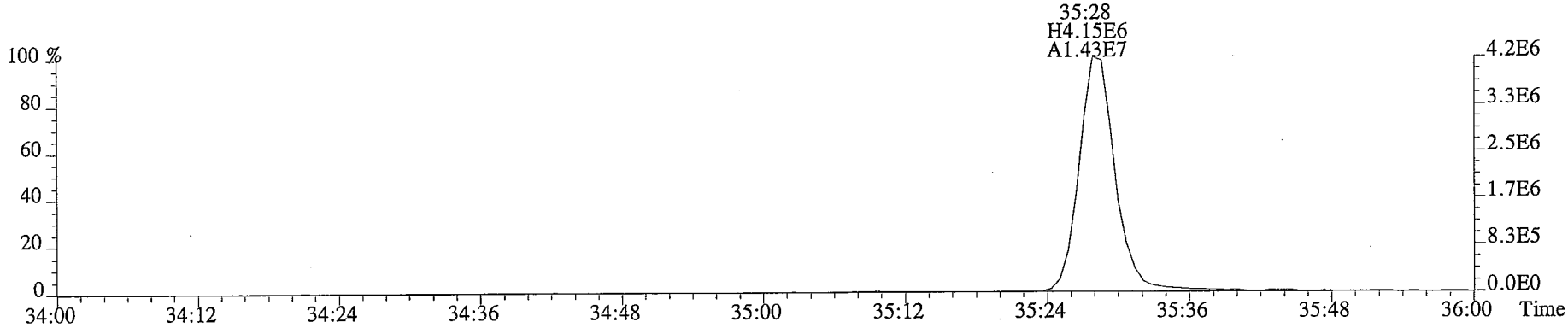
380.9760 S:5



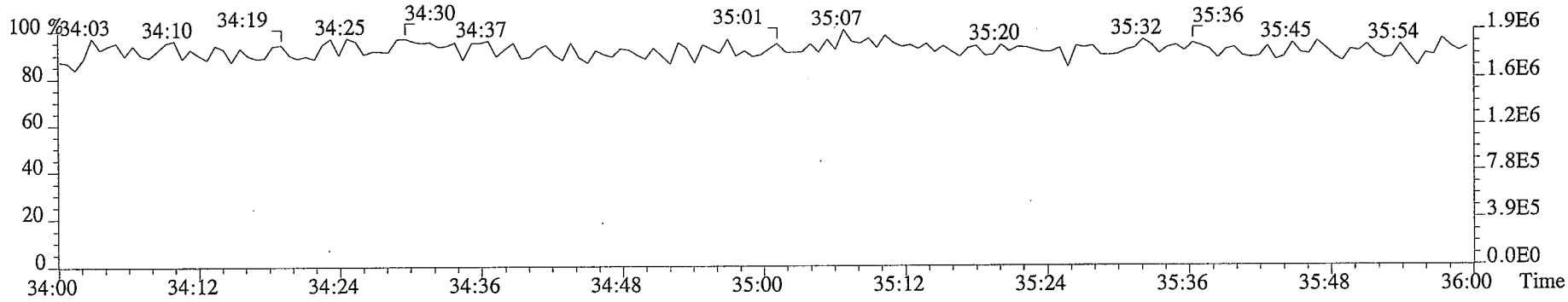
File:110721D2 #1-2305 Acq:22-JUL-2011 05:35:35 GC EI+ Voltage SIR Autospec-UltimaE
Sample#5 File Text:Vista Analytical Laboratory VG-7 Text:27592_3903_048 IPR-Soil-2 10g Exp:HCX_DB5
401.8560 S:5 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



403.8530 S:5 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



380.9760 S:5



Client ID: IPR-Soil-3 10g
Lab ID: 27592_3903_049

Filename: 110721D2 S:6 Acq:22-JUL-11 06:18:08
GC Column ID: db-5 ICal: HCXVG7-7-19-11 wt/vol:10.000

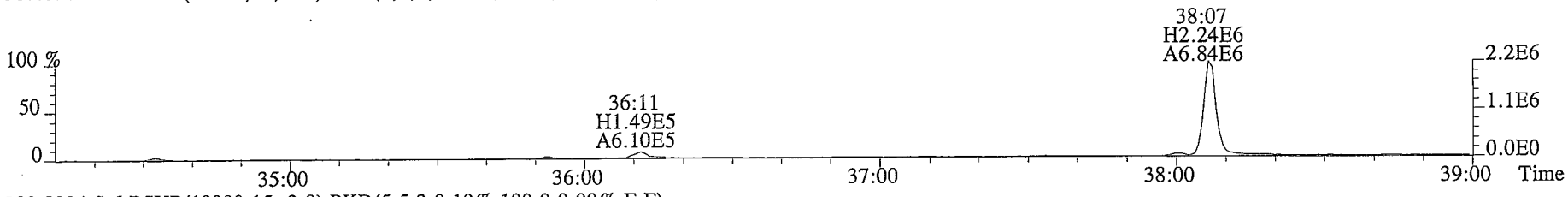
ConCal:
EndCAL:

	Name	Resp	RA	RRF	RT	Conc	Qualifiers	Rec	noise	Fac	DL
Unk	1,2,4,5,7,8-HCX	1.20e+07	1.34 y	0.14	38:07	411			*	2.5	*
IS	13C-1,2,3,7,8,9-HxCDF	4.08e+07	0.53 y	0.91	35:53	194		96.9			
C/Up	13C-1,2,3,7,8,9-HxCDD	3.21e+07	1.31 y	0.71	35:28	197		98.5			
RS/R	13C-1,2,3,4,6,9-HxCDF	4.60e+07	0.53 y	1.00	34:33	200					

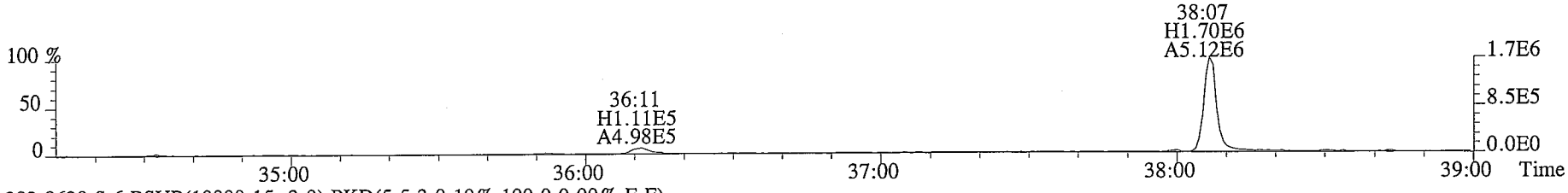
Analyst: MJ

Date: 7/22/11

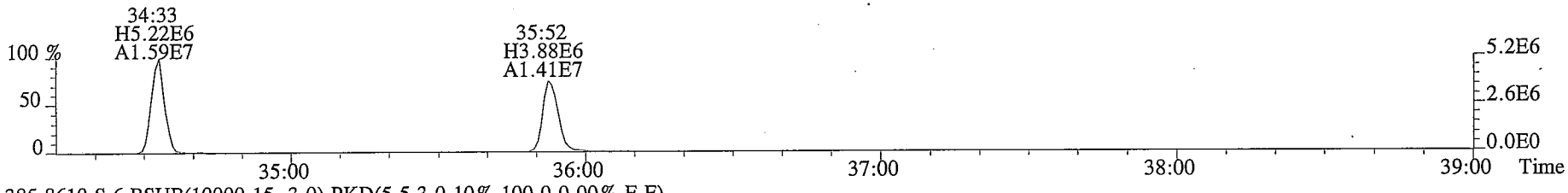
File:110721D2 #1-2305 Acq:22-JUL-2011 06:18:08 GC EI+ Voltage SIR Autospec-UltimaE
Sample#6 File Text:Vista Analytical Laboratory VG-7 Text:27592_3903_049 IPR-Soil-3 10g Exp:HCX_DB5
387.8364 S:6 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



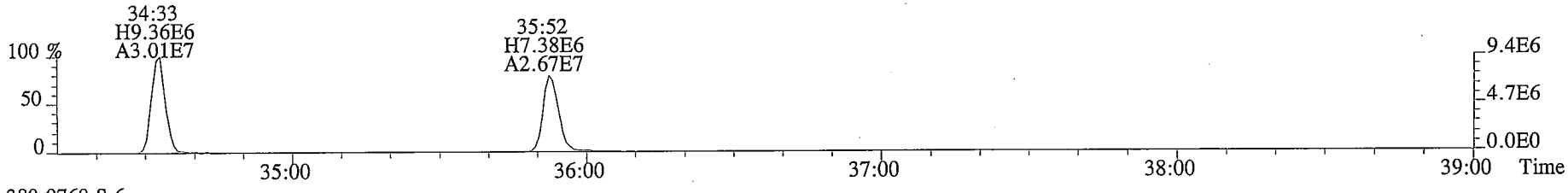
389.8334 S:6 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



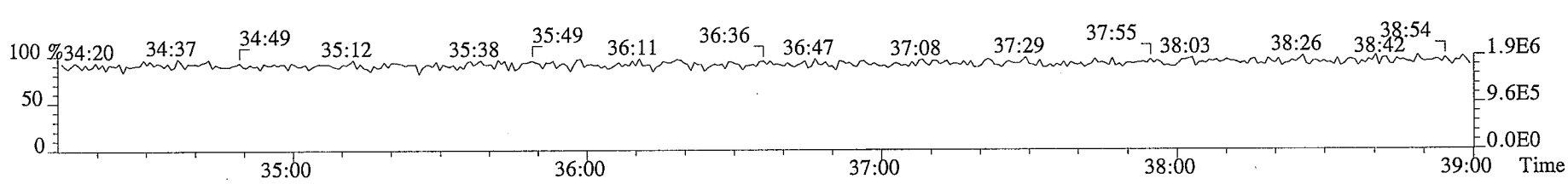
383.8639 S:6 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



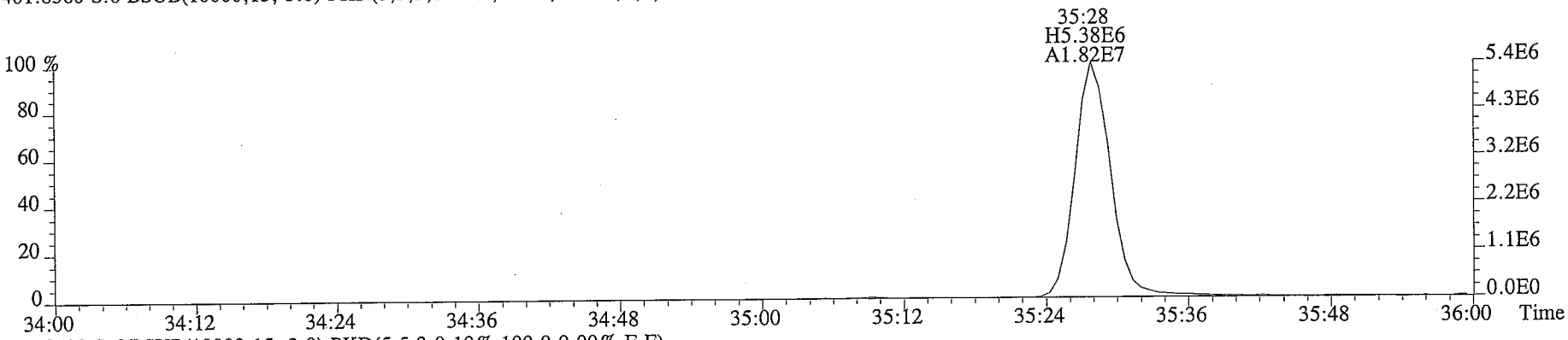
385.8610 S:6 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



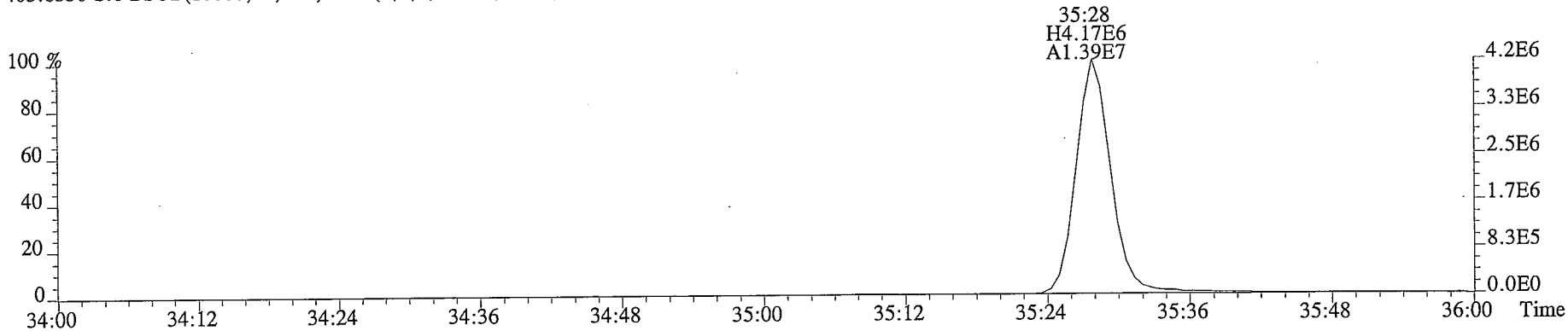
380.9760 S:6



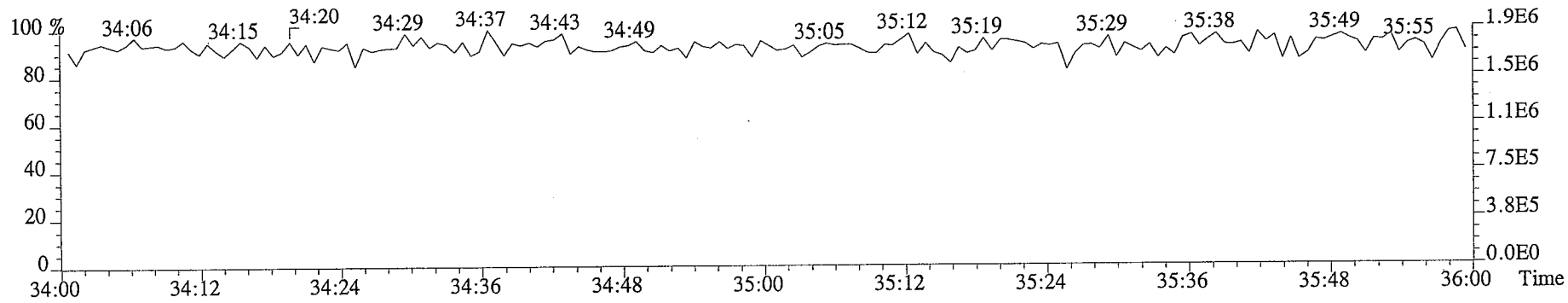
File:110721D2 #1-2305 Acq:22-JUL-2011 06:18:08 GC EI+ Voltage SIR Autospec-UltimaE
Sample#6 File Text:Vista Analytical Laboratory VG-7 Text:27592_3903_049 IPR-Soil-3 10g Exp:HCX_DB5
401.8560 S:6 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



403.8530 S:6 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



380.9760 S:6



Client ID: IPR-Soil-4 10g Filename: 110721D2 S:7 Acq:22-JUL-11 07:00:40 ConCal:
Lab ID: 27592_3903_050 GC Column ID: db-5 ICal: HCXVG7-7-19-11 wt/vol:10.000 EndCAL:

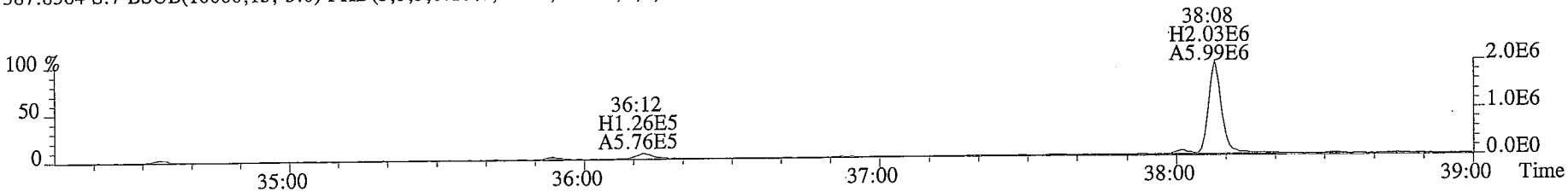
	Name	Resp	RA	RRF	RT	Conc	Qualifiers	Rec	noise	Fac	DL
Unk	1,2,4,5,7,8-HCX	1.05e+07	1.30 y	0.14	38:08	386			*	2.5	*
IS	13C-1,2,3,7,8,9-HxCDF	3.83e+07	0.52 y	0.91	35:54	191		95.4			
C/Up	13C-1,2,3,7,8,9-HxCDD	3.07e+07	1.28 y	0.71	35:29	197		98.7			
RS/R	13C-1,2,3,4,6,9-HxCDF	4.38e+07	0.53 y	1.00	34:34	200					

Analyst: MS

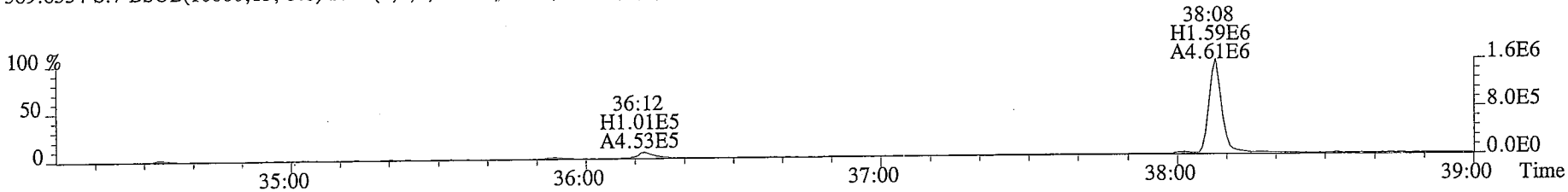
Date: 7/22/11

File:110721D2 #1-2306 Acq:22-JUL-2011 07:00:40 GC EI+ Voltage SIR Autospec-UltimaE
Sample#7 File Text:Vista Analytical Laboratory VG-7 Text:27592_3903_050 IPR-Soil-4 10g Exp:HCX_DB5

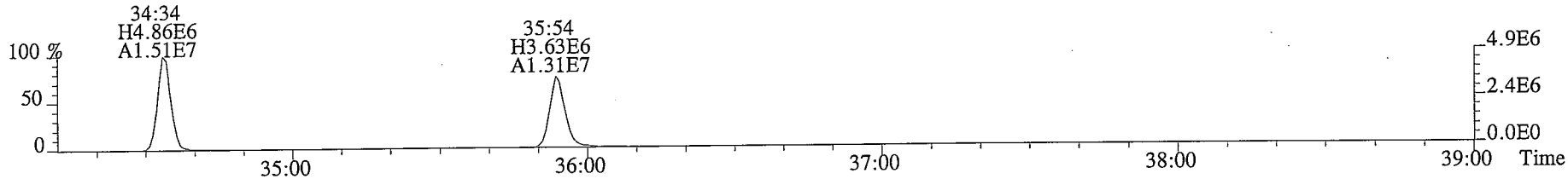
387.8364 S:7 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



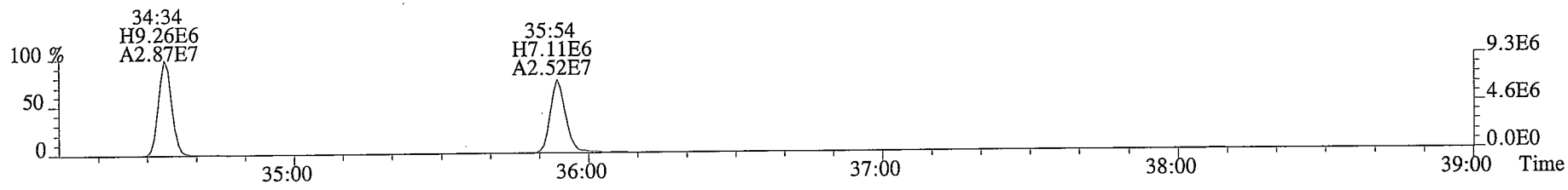
389.8334 S:7 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



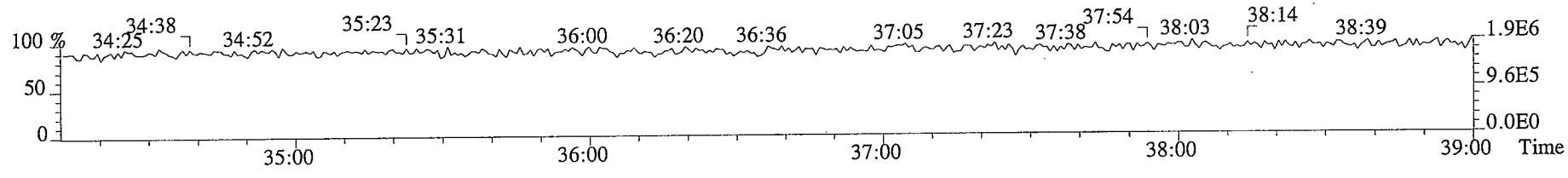
383.8639 S:7 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



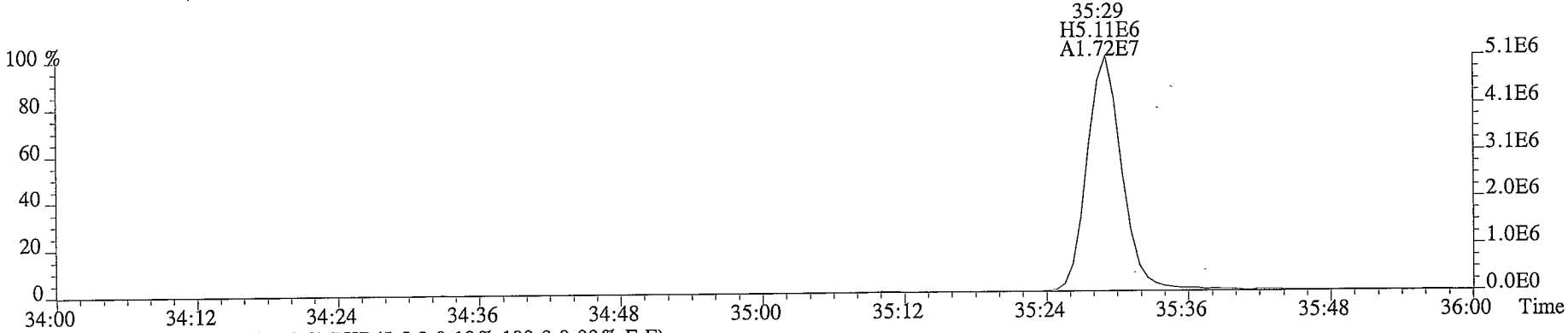
385.8610 S:7 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



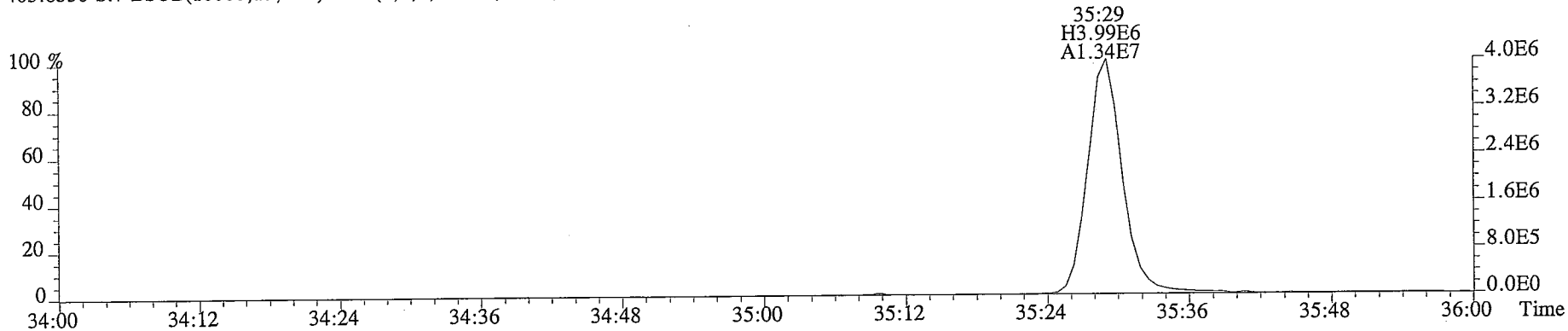
380.9760 S:7



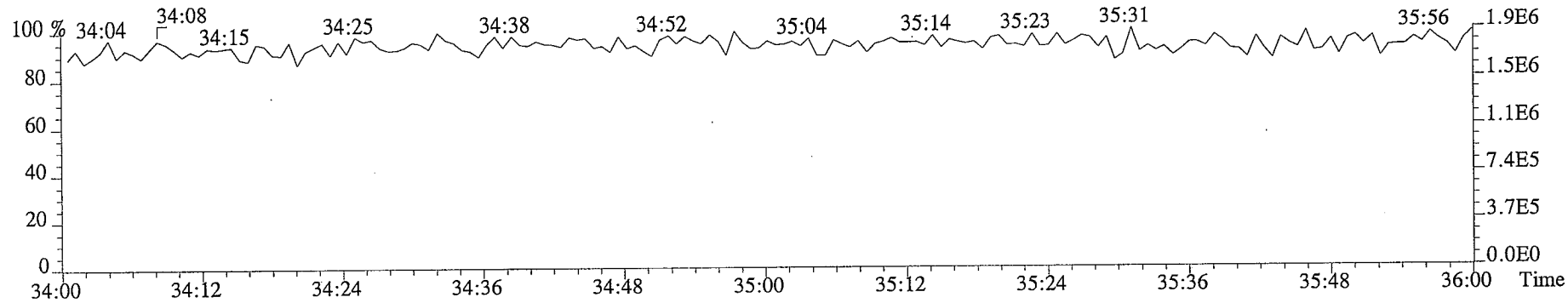
File:110721D2 #1-2306 Acq:22-JUL-2011 07:00:40 GC EI+ Voltage SIR Autospec-UltimaE
Sample#7 File Text:Vista Analytical Laboratory VG-7 Text:27592_3903_050 IPR-Soil-4 10g Exp:HCX_DB5
401.8560 S:7 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



403.8530 S:7 BSUB(10000,15,-3.0) PKD(5,5,3,0.10%,100.0,0.00%,F,F)



380.9760 S:7



Attachment D

IPR Study Liquid (Full Laboratory Deliverable)

IPR
Hexachloroxanthene

Vista Analytical Laboratory

Hexachloroxanthene IPR

Analyte	Spike (pg/L)	IPR1	IPR2	IPR3	IPR4	Ave	Method Limits	%RSD	Method Criteria
1,2,4,5,7,8-HCX	4000	3340	2770	2780	3170	3015	2400-5600	9.5	40.0
^{13}C-1,2,3,7,8,9-HxCDF	2000	1810	1790	1840	1990	1858	700-2700	4.9	50.0
^{13}C-1,2,3,7,8,9-HxCDD	2000	2020	2010	2100	2130	2065	700-2700	2.9	50.0
^{13}C-1,2,3,4,6,9-HxCDF	2000	2000	2000	2000	2000	2000	700-2700	0.0	50.0
1,2,4,5,7,8-HxCX	**%Rec**	83.5	69.3	69.5	79.3	75.4	60-140	9.5	40.0
^{13}C-1,2,3,7,8,9-HxCDF	**%Rec**	90.5	89.5	92.0	99.5	92.9	35-135	4.9	50.0
^{13}C-1,2,3,7,8,9-HxCDD	**%Rec**	101.0	100.5	105.0	106.5	103.3	35-135	2.9	50.0

Matrix: Aqueous
Chemist: CV
7/31/2011
Batch: 3923
using IPR limits from 1668A

PROCESS SHEET

Project No.-AR: 27592-252 of 257

Prep Due: 7/16/2011 **Project Due:** 7/14/2011

Hold Due: 3/8/2012 **TAT:** 21

Client: Vista Analytical Laboratory(AALCA01D)

Client Manager: Christina A. Vredevoe

Method: Hexachlorophene | Hexachlorophene
Hexachloroxanthene | Hexachloroxanthene

Split Type:

3923

Matrix: Aqueous

LabID	Recon	Client-ID	Description	Date Received	SLoc	Shelf
043	☐	IPR-Aqueous-1		3/9/2011		
044	☐	IPR-Aqueous-2		3/9/2011		
045	☐	IPR-Aqueous-3		3/9/2011		
046	☐	IPR-Aqueous-4		3/9/2011		

Instructions:

HCX only - no split

Report Options

Report Level: TEQ Type:

EDD Type:

Report Group: Dioxins NoMDL No %Solid

Samples Reconciled By: N/A / /

Vial Box ID: _____

Date Requested 7/3/2011

Project: 27592

Extraction Set: 3923

Chemist: C. Vredenve 7/31/11

Method(s): Hexachlorophene/Hexachloroxanthene | Hexachlorophene/Hexachloroxanthene

Prep time: 1019

C	VISTA Sample ID	G Eqv	Sample Amt. (L)	IS/NS CHEM/ WIT DATE	CRS CHEM/WIT DATE	AP CHEM/Date	~~ABSG~~ SG CHEM/Date	AA CHEM/Date	Florisil CHEM/Date	RS CHEM/WIT DATE
☐	0_3923_MB001	N/A	(1.000)	CV AR 7/31/11	CV AR 7/31/11	N/A	CV 7/31/11	N/A	N/A	CV AR 7/31/11
☐	27592_3923_043	↓	↓	↓	↓	↓	↓	↓	↓	↓
☐	27592_3923_044									
☐	27592_3923_045									
☐	27592_3923_046									

IS Name	NS Name	CRS Name	RS Name
PCDD/F	PCDD/F	PCDD/F	PCDD/F
PCB	PCB	PCB	PCB
HCx 110706A 10µL	HCx 110701A 10µL	HCx 110503B 10µL	HCx 110503C 10µL

Cycle Time
Start: N/A
Stop: ↓

APP.: (SEFUN) SOX SDS
SOLV: DCM
Other: N/A
Final Volume(s): 20µL / CHx

Check Out:
Chemist: N/A / /
Check-In:
Chemist: ↓ / /
Balance ID: ↓

Comments:

CALIBRATION STANDARDS REVIEW CHECKLIST

Vista Analytical Laboratory

Beg. Calibration ID: ST110802F2-1

End Calibration ID: N/A

	Beg.	End
Ion abundance within QC limits?	✓	NA
Concentration within range?	✓	✓
First and last eluters present?	NA	
Retention Times within criteria?	NA	
Verification Std. named correctly? (ST-Year-Month-Day-VG ID)	✓	
Forms signed and dated?	✓	
Correct ICAL referenced?	✓	
Run Log:		
-Data file matches Conc Cal ID?	✓	
-Correct instrument listed?	✓	
-Samples within 12-hour clock?	(y)	n

	Beg.	End
Mass resolution > 10,000? ▪ Method 1614 > 5,000; CARB 429 > 8,000	✓	✓
TCDD/TCDF valleys < 25%?	NA	N/A
Peaks integrated correctly?	✓	
Manual integrations included?		
8280 CS1 Ending Standard		
-Ratios within limits		
-S/N > 2.5:1		
-CS1 within 12-hour clock		✓

Comments:

Reviewed by: DMS 8/3/11
Initials & Date

** Ending standard criteria applicable to 8290 only.*

Vista Analytical Laboratory VG-9

Dataset: Untitled

Last Altered: Wednesday, August 03, 2011 12:15:31 PM Pacific Standard Time
Printed: Wednesday, August 03, 2011 12:15:52 PM Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-8-01-11.cdb 02 Aug 2011 09:02:25

Compound name: 1,2,4,5,7,8-HCX

	Name	ID	Acq.Date	Acq.Time
1	110802F2_1	ST110802F2-1 HCX CS3 110706E	02-Aug-11	15:07:04
2	110802F2_2	SOLVENT BLANK	02-Aug-11	15:49:08
3	110802F2_3	0_3922_MB001	02-Aug-11	16:32:46
4	110802F2_4	0_3923_MB001	02-Aug-11	17:16:23
5	110802F2_5	27592_3922_001 MDL-AQ-1 1	02-Aug-11	18:00:02
6	110802F2_6	27592_3922_002 MDL-AQ-2 1	02-Aug-11	18:43:40
7	110802F2_7	27592_3922_003 MDL-AQ-3 1	02-Aug-11	19:27:13
8	110802F2_8	27592_3922_004 MDL-AQ-4 1	02-Aug-11	20:10:51
9	110802F2_9	27592_3922_005 MDL-AQ-5 1	02-Aug-11	20:54:18
10	110802F2_10	27592_3922_006 MDL-AQ-6 1	02-Aug-11	21:37:56
11	110802F2_11	27592_3922_007 MDL-AQ-7 1	02-Aug-11	22:21:34
12	110802F2_12	27592_3923_043 IPR-Aqueous-1 1	02-Aug-11	23:05:08
13	110802F2_13	27592_3923_044 IPR-Aqueous-2 1	02-Aug-11	23:48:41
14	110802F2_14	27592_3923_045 IPR-Aqueous-3 1	03-Aug-11	00:32:14
15	110802F2_15	27592_3923_046 IPR-Aqueous-4 1	03-Aug-11	01:15:51
16	110802F2_16	SOLVENT BLANK	03-Aug-11	01:59:29

CALIBRATION STANDARDS REVIEW CHECKLIST

Vista Analytical Laboratory

Beg. Calibration ID: ST110801F3-1 End Calibration ID: NA

	Beg.	End
Ion abundance within QC limits?	✓	NA
Concentration within range?	✓	↓
First and last eluters present?	NA	NA
Retention Times within criteria?	NA	NA
Verification Std. named correctly? (ST-Year-Month-Day-VG ID)	✓	
Forms signed and dated?	✓	
Correct ICAL referenced?	✓	
Run Log:		
-Data file matches Conc Cal ID?	✓	
-Correct instrument listed?	✓	↓
-Samples within 12-hour clock?	(y)	n

	Beg.	End
Mass resolution ~~> 10,000?~~	✓	✓
▪ Method 1614 > 5,000; CARB 429 > 8,000		
TCDD/TCDF valleys < 25%?	NA	NA
Peaks integrated correctly?		
Manual integrations included?		
8280 CS1 Ending Standard		
-Ratios within limits		
-S/N > 2.5:1		
-CS1 within 12-hour clock		↓

Comments:

Reviewed by: 8/3/11
Initials & Date

* *Ending standard criteria applicable to 8290 only.*

Quantify Compound Summary Report **MassLynx 4.1**
Vista Analytical Laboratory VG-9

Dataset: Untitled

Last Altered: Wednesday, August 03, 2011 1:36:16 PM Pacific Standard Time
Printed: Wednesday, August 03, 2011 1:36:54 PM Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-8-01-11.cdb 02 Aug 2011 09:02:25

Compound name: 1,2,4,5,7,8-HCX

	Name	ID	Acq.Date	Acq.Time
1	110801F3_1	ST110801F3-1 HCX CS3 110706E	02-Aug-11	00:42:21
2	110801F3_2	27592_3923_043 IPR-Aqueous-1 1	02-Aug-11	01:24:27
3	110801F3_3	27592_3923_044 IPR-Aqueous-2 1	02-Aug-11	02:07:59
4	110801F3_4	27592_3923_045 IPR-Aqueous-3 1	02-Aug-11	02:51:32
5	110801F3_5	27592_3923_046 IPR-Aqueous-4 1	02-Aug-11	03:35:05

Dataset: C:\MassLynx\Default.pro\Results\110802F2\110802F2_4.qld

Last Altered: Wednesday, August 03, 2011 12:13:08 Pacific Standard Time
Printed: Wednesday, August 03, 2011 12:13:48 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-8-01-11.cdb 02 Aug 2011 09:02:25

Name: 110802F2_4, Date: 02-Aug-2011, Time: 17:16:23, ID: 0_3923_MB001, Description: 0_3923_MB001

	# Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1 1,2,4,5,7,8-HCX				0.220	1.000				8.03
2	2 13C-1,2,3,7,8,9-HxCDF	1.17e6	0.53	NO	0.869	1.000	34.96	1882.0	94.1	2.47
3	3 13C-1,2,3,7,8,9-HxCDD	1.07e6	1.26	NO	0.717	1.000	34.55	2095.2	105	3.04
4	4 13C-1,2,3,4,6,9-HxCDF	1.43e6	0.52	NO	1.00	1.000	33.65	2000.0	100	2.15

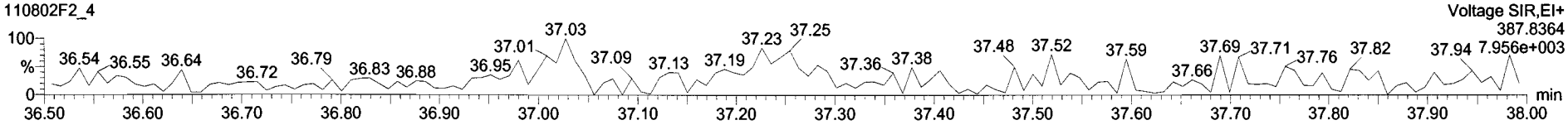
FEB 8/3/11

Dataset: Untitled

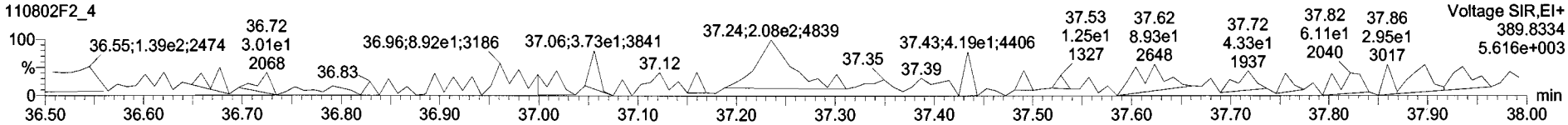
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_4, Date: 02-Aug-2011, Time: 17:16:23, ID: 0_3923_MB001, Description: 0_3923_MB001

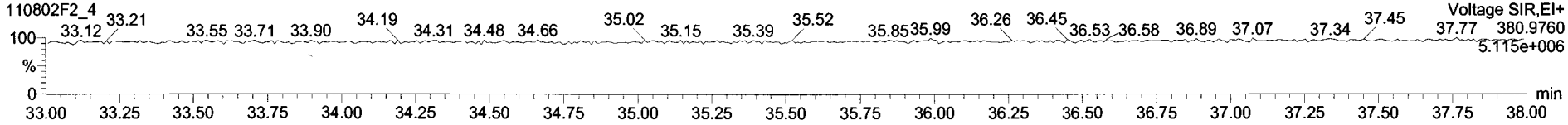
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1

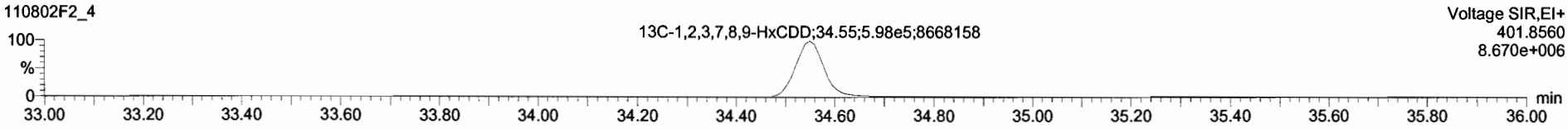


Dataset: Untitled

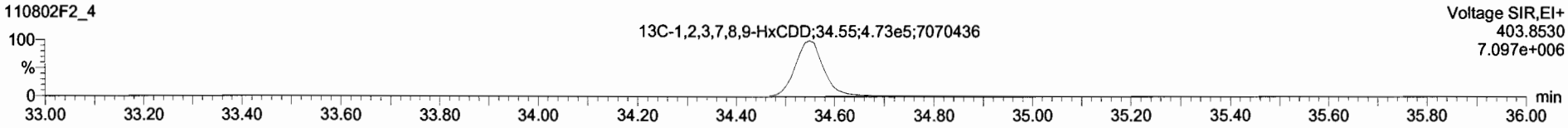
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_4, Date: 02-Aug-2011, Time: 17:16:23, ID: 0_3923_MB001, Description: 0_3923_MB001

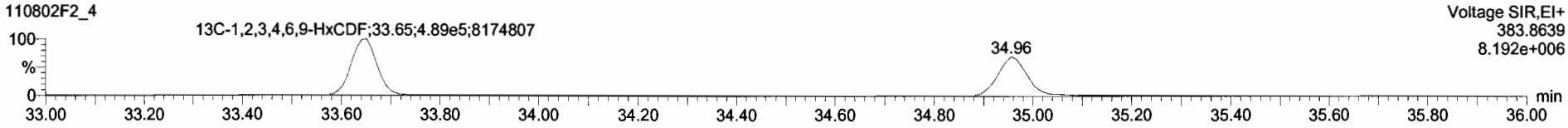
13C-1,2,3,7,8,9-HxCDD



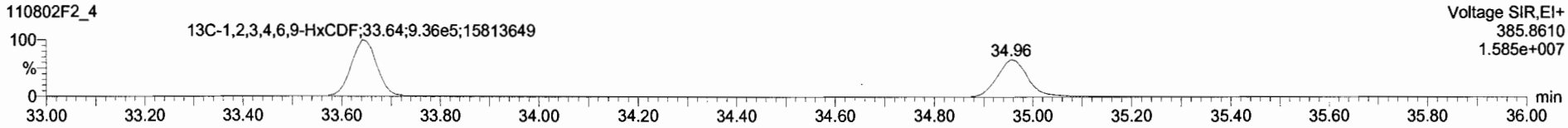
13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,4,6,9-HxCDF



13C-1,2,3,4,6,9-HxCDF



Dataset: C:\MassLynx\Default.pro\Results\110802F2\110802F2_12.qld

Last Altered: Wednesday, August 03, 2011 12:53:07 Pacific Standard Time
Printed: Wednesday, August 03, 2011 12:53:45 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-8-01-11.cdb 02 Aug 2011 09:02:25

Name: 110802F2_12, Date: 02-Aug-2011, Time: 23:05:08, ID: 27592_3923_043 IPR-Aqueous-1 1, Description: IPR-Aqueous-1

	# Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1 1,2,4,5,7,8-HCX	3.29e5	1.25	NO	0.220	1.000	37.24	2969.5		12.2
2	2 13C-1,2,3,7,8,9-HxCDF	1.01e6	0.53	NO	0.869	1.000	34.97	1800.4	90.0	2.62
3	3 13C-1,2,3,7,8,9-HxCDD	9.74e5	1.24	NO	0.717	1.000	34.55	2111.4	106	3.24
4	4 13C-1,2,3,4,6,9-HxCDF	1.29e6	0.51	NO	1.00	1.000	33.65	2000.0	100	2.28

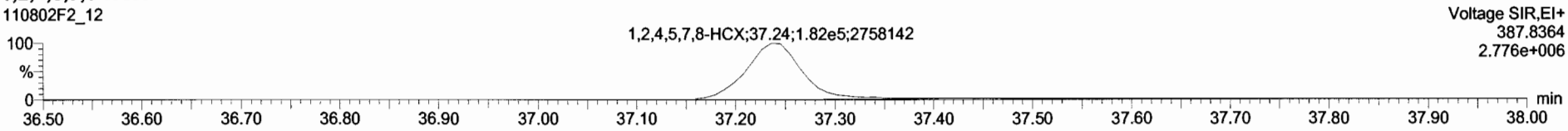
FEB 8/3/11

Dataset: Untitled

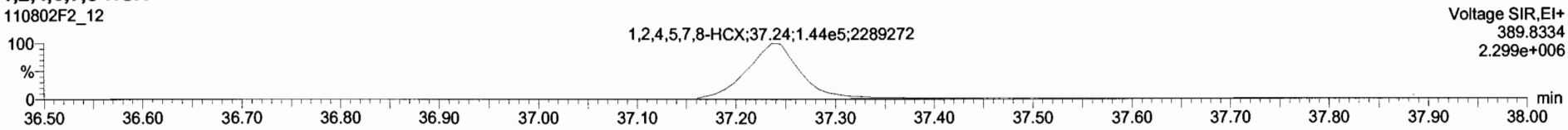
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_12, Date: 02-Aug-2011, Time: 23:05:08, ID: 27592_3923_043 IPR-Aqueous-1 1, Description: IPR-Aqueous-1

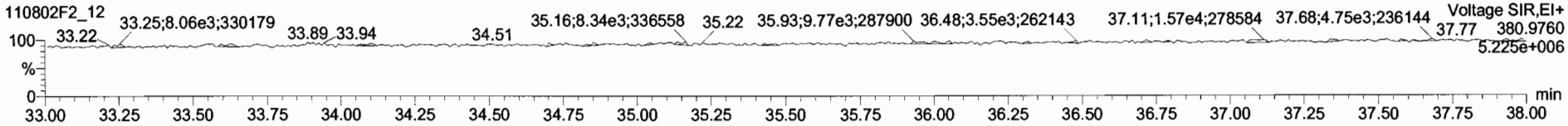
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1



TargetLynx - untitled - [Chromatogram]

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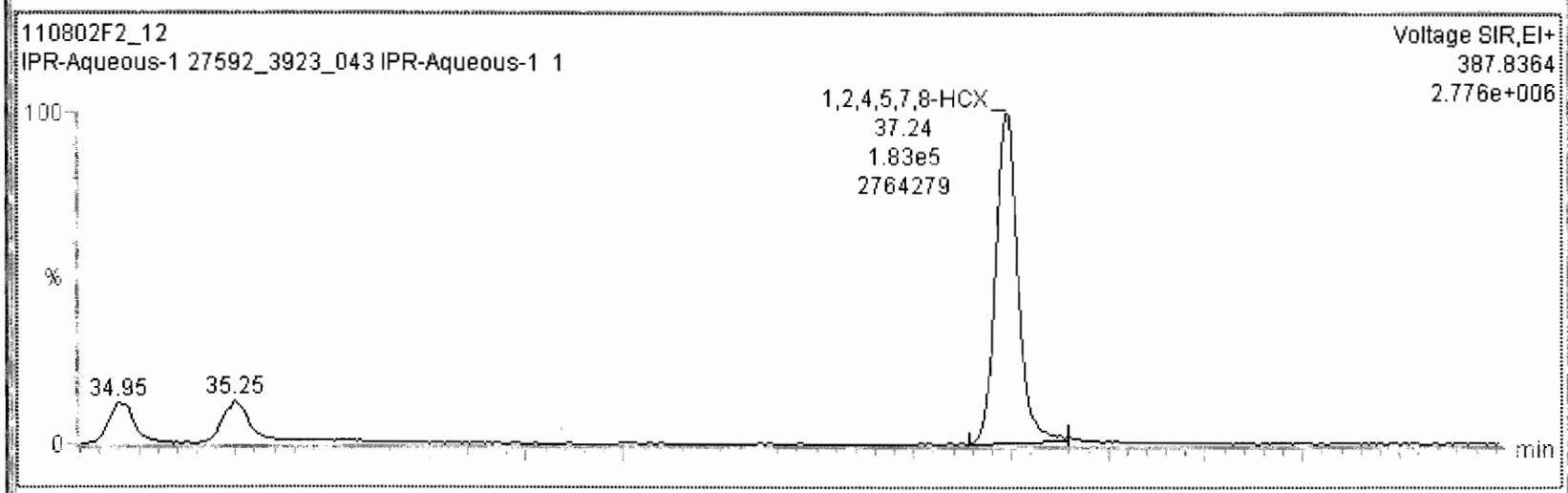
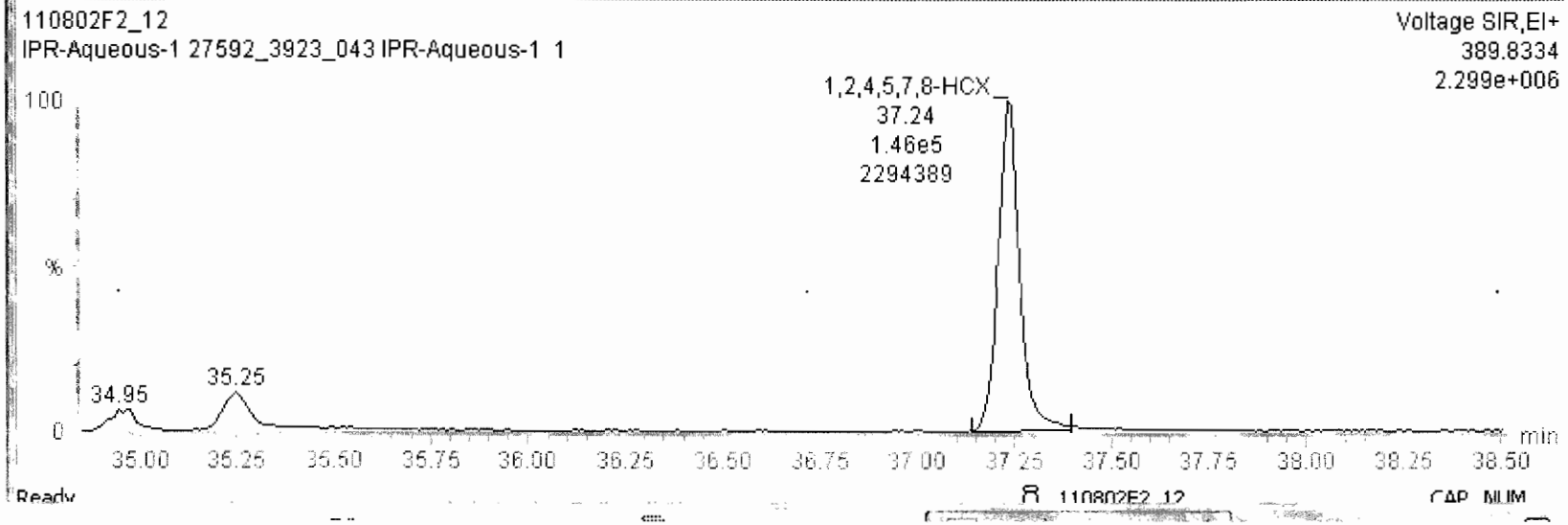
110802F2_12 - 27592_3923_043 IPR-Aqueous-1 1 - IPR-Aqueous-1

	#	Name	Resp	RA	n/y	RRF	wt/vol	RT	Conc.	%Rec	DL
1	1	1,2,4,5,7,8-HCX	3.29e5	1.25	NO	0.22	1.000	37.24	2970		12.2
2	2	13C-1,2,3,7,8,9-HxCDF	1.01e6	0.53	NO	0.87	1.000	34.97	1800	90.0	2.62
3	3	13C-1,2,3,7,8,9-HxCDD	9.74e5	1.24	NO	0.72	1.000	34.55	2110	106	3.24
4	4	13C-1,2,3,4,6,9-HxCDF	1.29e6	0.51	NO	1.00	1.000	33.65	2000	100	2.28
5	5	PFK1					1.000				





Ready

110802F2_12

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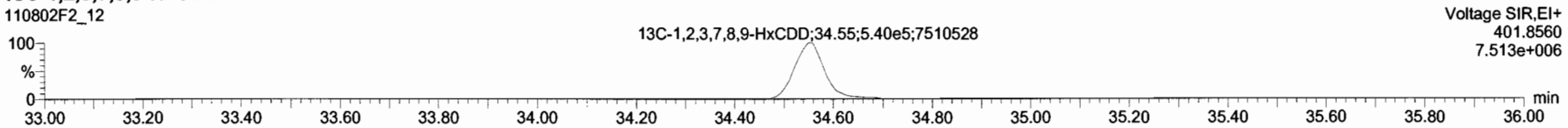
Dataset: Untitled

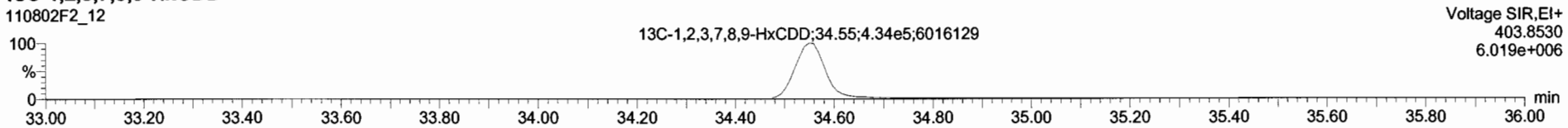
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_12, Date: 02-Aug-2011, Time: 23:05:08, ID: 27592_3923_043 IPR-Aqueous-1 1, Description: IPR-Aqueous-1

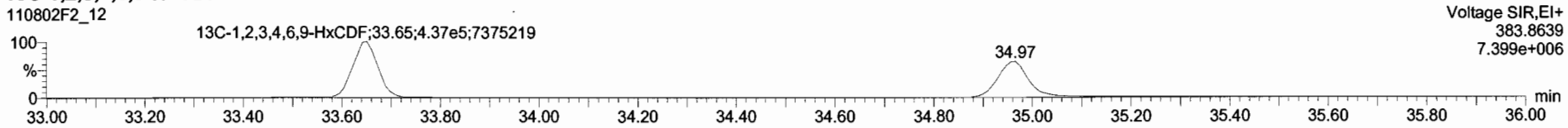
13C-1,2,3,7,8,9-HxCDD



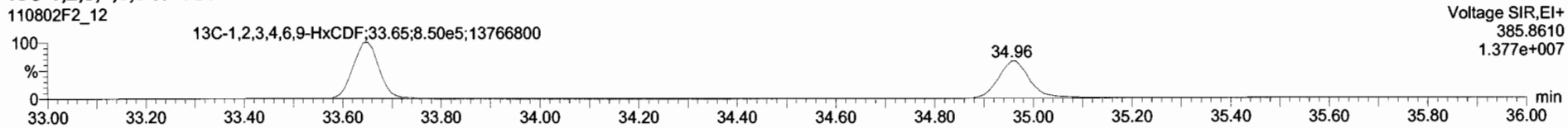
13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,4,6,9-HxCDF



13C-1,2,3,4,6,9-HxCDF



Dataset: C:\MassLynx\Default.pro\Results\110802F2\110802F2_13.qld

Last Altered: Wednesday, August 03, 2011 12:55:21 Pacific Standard Time
Printed: Wednesday, August 03, 2011 12:57:02 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-8-01-11.cdb 02 Aug 2011 09:02:25

Name: 110802F2_13, Date: 02-Aug-2011, Time: 23:48:41, ID: 27592_3923_044 IPR-Aqueous-2 1, Description: IPR-Aqueous-2

	# Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1 1,2,4,5,7,8-HCX	2.87e5	1.30	NO	0.220	1.000	37.24	2668.8		15.3
2	2 13C-1,2,3,7,8,9-HxCDF	9.78e5	0.52	NO	0.869	1.000	34.97	1718.7	85.9	2.59
3	3 13C-1,2,3,7,8,9-HxCDD	1.00e6	1.26	NO	0.717	1.000	34.56	2130.8	107	2.72
4	4 13C-1,2,3,4,6,9-HxCDF	1.31e6	0.53	NO	1.00	1.000	33.65	2000.0	100	2.25

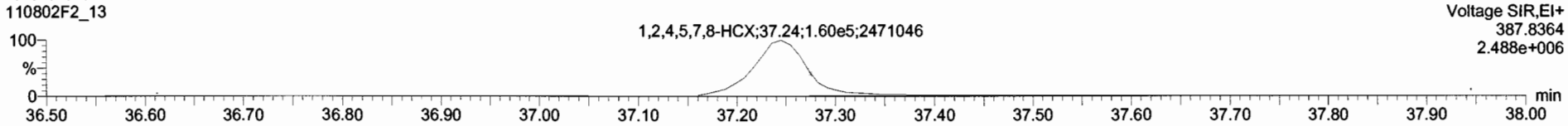
FEB 8/3/11

Dataset: Untitled

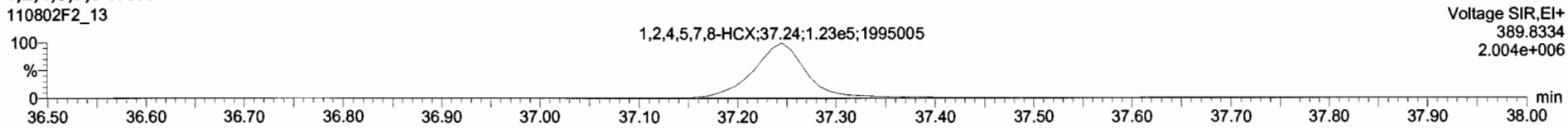
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_13, Date: 02-Aug-2011, Time: 23:48:41, ID: 27592_3923_044 IPR-Aqueous-2 1, Description: IPR-Aqueous-2

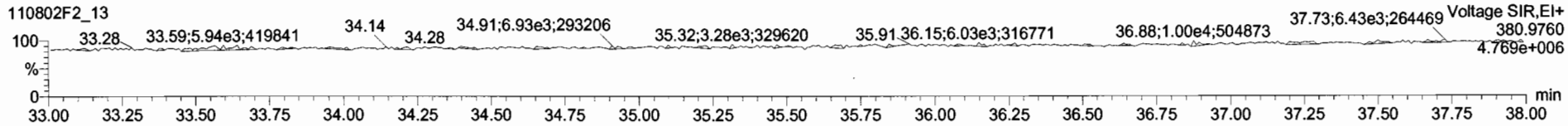
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1



TargetLynx - Untitled * - [Chromatogram]

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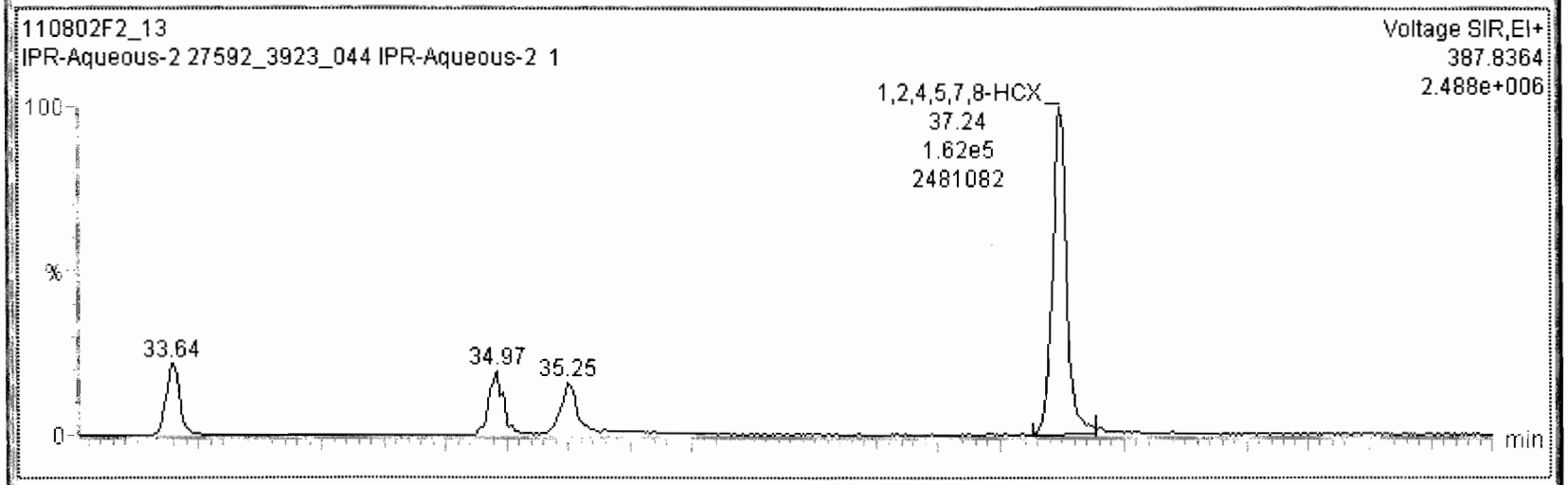
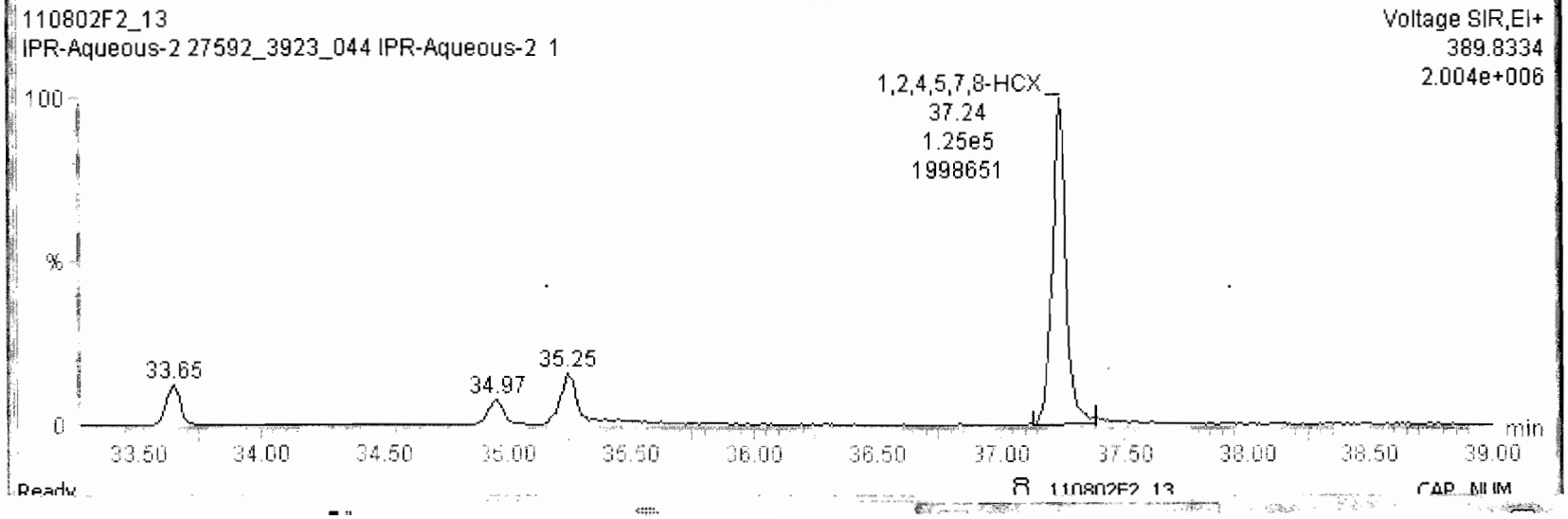
110802F2_13 - 27592_3923_044 IPR-Aqueous-2 1 - IPR-Aqueous-2

	#	Name	Resp	RA	n/y	RRF	wt/vol	RT	Conc.	%Rec	DL
1	1	1,2,4,5,7,8-HCX	2.87e5	1.30	NO	0.22	1.000	37.24	2670		15.3
2	2	13C-1,2,3,7,8,9-HxCDF	9.78e5	0.52	NO	0.87	1.000	34.97	1720	85.9	2.59
3	3	13C-1,2,3,7,8,9-HxCDD	1.00e6	1.26	NO	0.72	1.000	34.56	2130	107	2.72
4	4	13C-1,2,3,4,6,9-HxCDF	1.31e6	0.53	NO	1.00	1.000	33.65	2000	100	2.25
5	5	PFK1					1.000				



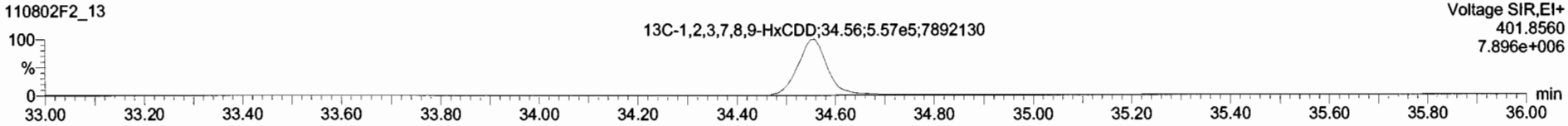


Dataset: Untitled

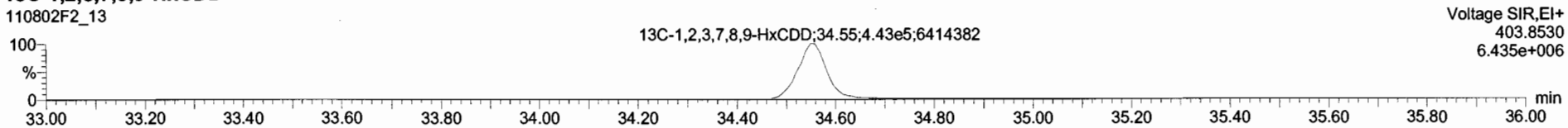
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_13, Date: 02-Aug-2011, Time: 23:48:41, ID: 27592_3923_044 IPR-Aqueous-2 1, Description: IPR-Aqueous-2

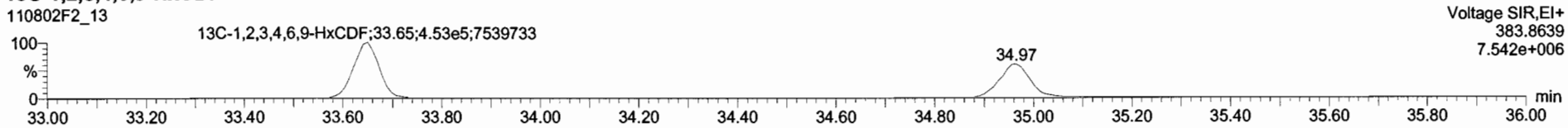
13C-1,2,3,7,8,9-HxCDD



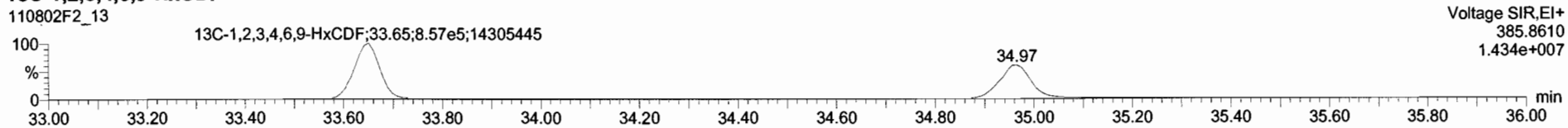
13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,4,6,9-HxCDF



13C-1,2,3,4,6,9-HxCDF



Dataset: C:\MassLynx\Default.pro\Results\110802F2\110802F2_14.qld

Last Altered: Wednesday, August 03, 2011 12:58:30 Pacific Standard Time
Printed: Wednesday, August 03, 2011 12:59:14 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-8-01-11.cdb 02 Aug 2011 09:02:25

Name: 110802F2_14, Date: 03-Aug-2011, Time: 00:32:14, ID: 27592_3923_045 IPR-Aqueous-3 1, Description: IPR-Aqueous-3

	# Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1 1,2,4,5,7,8-HCX	3.43e5	1.27	NO	0.220	1.000	37.24	2958.9		15.0
2	2 13C-1,2,3,7,8,9-HxCDF	1.05e6	0.52	NO	0.869	1.000	34.97	1844.3	92.2	2.62
3	3 13C-1,2,3,7,8,9-HxCDD	1.01e6	1.24	NO	0.717	1.000	34.55	2150.2	108	3.19
4	4 13C-1,2,3,4,6,9-HxCDF	1.32e6	0.52	NO	1.00	1.000	33.65	2000.0	100	2.27

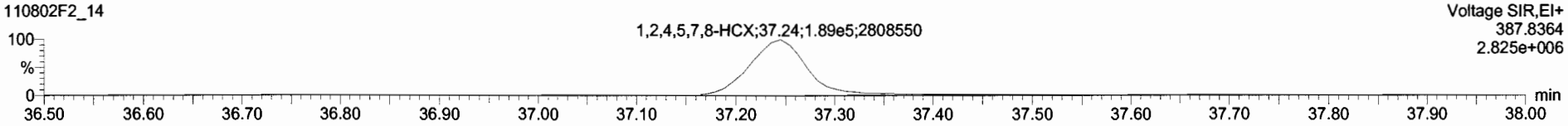
Feb 8/3/11

Dataset: Untitled

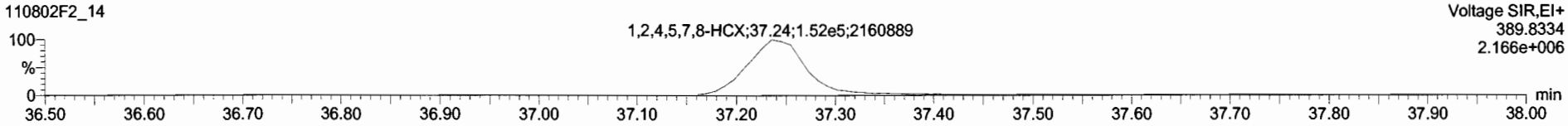
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_14, Date: 03-Aug-2011, Time: 00:32:14, ID: 27592_3923_045 IPR-Aqueous-3 1, Description: IPR-Aqueous-3

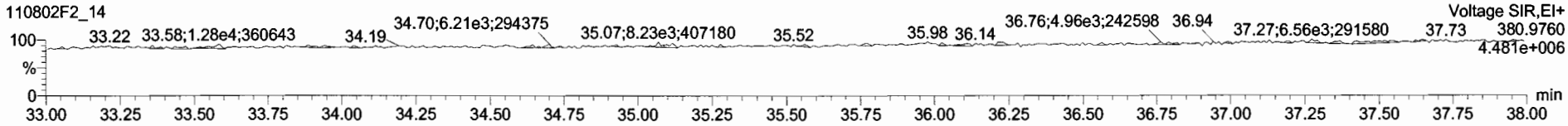
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1



TargetLynx - untitled - [Chromatogram]

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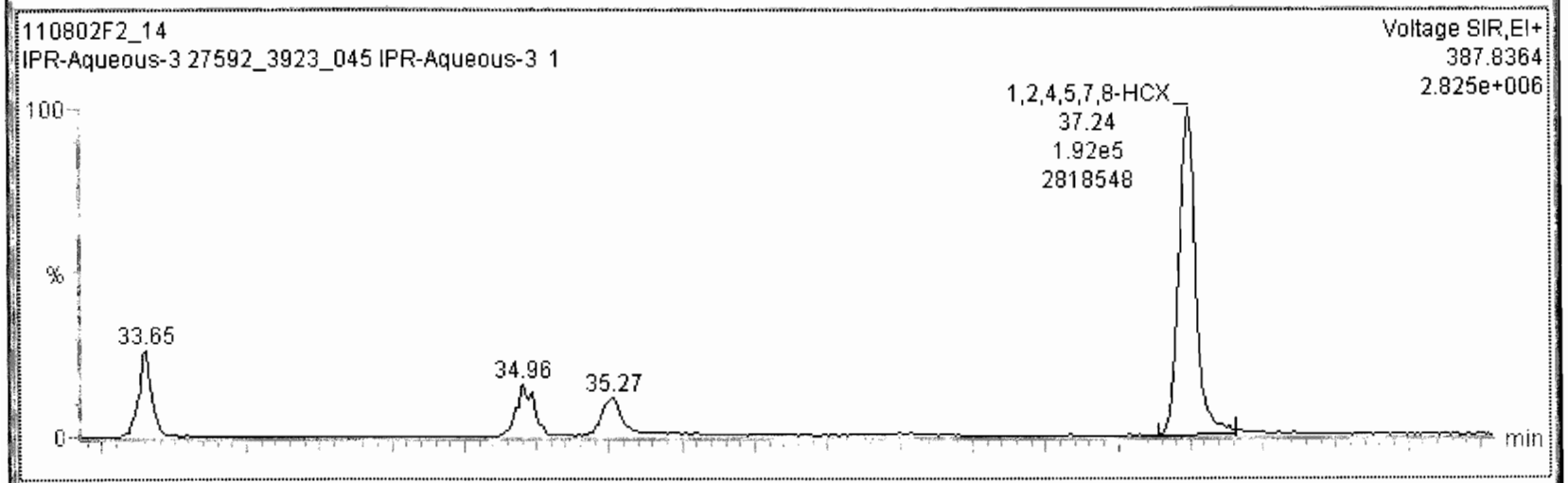
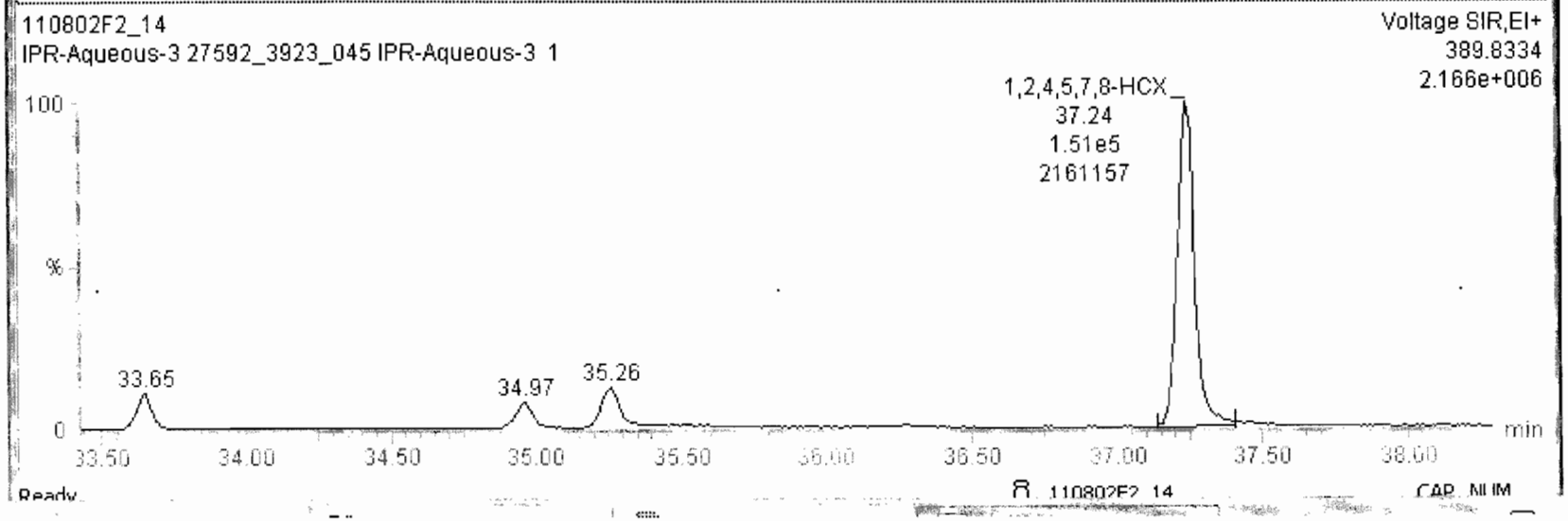
110802F2_14 - 27592_3923_045 IPR-Aqueous-3 1 - IPR-Aqueous-3

	#	Name	Resp	RA	n/y	RRF	wt/vol	RT	Conc.	%Rec	DL
1	1	1,2,4,5,7,8-HCX	3.43e5	1.27	NO	0.22	1.000	37.24	2960		15.0
2	2	13C-1,2,3,7,8,9-HxCDF	1.05e6	0.52	NO	0.87	1.000	34.97	1840	92.2	2.62
3	3	13C-1,2,3,7,8,9-HxCDD	1.01e6	1.24	NO	0.72	1.000	34.55	2150	108	3.19
4	4	13C-1,2,3,4,6,9-HxCDF	1.32e6	0.52	NO	1.00	1.000	33.65	2000	100	2.27
5	5	PFK1					1.000				



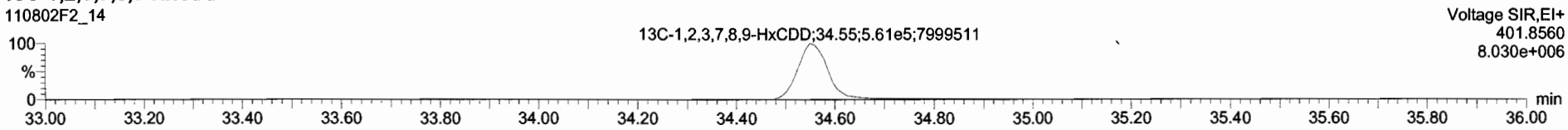


Dataset: Untitled

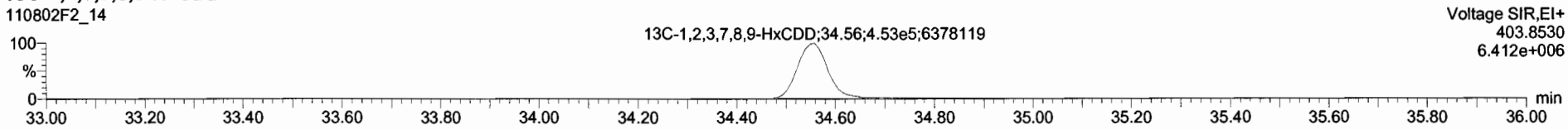
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_14, Date: 03-Aug-2011, Time: 00:32:14, ID: 27592_3923_045 IPR-Aqueous-3 1, Description: IPR-Aqueous-3

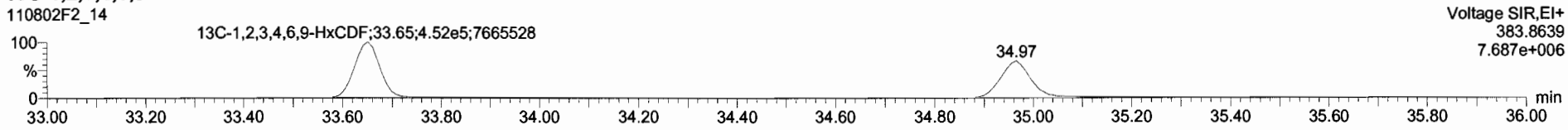
13C-1,2,3,7,8,9-HxCDD



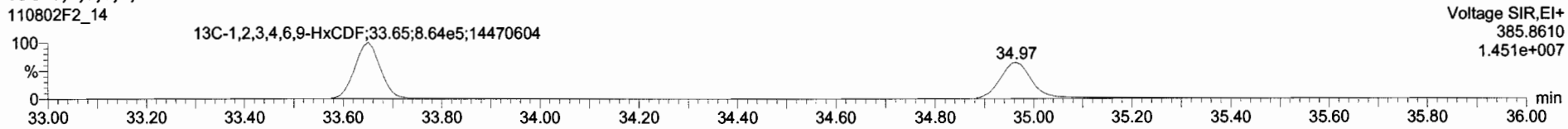
13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,4,6,9-HxCDF



13C-1,2,3,4,6,9-HxCDF



Dataset: C:\MassLynx\Default.pro\Results\110802F2\110802F2_15.qld

Last Altered: Wednesday, August 03, 2011 13:00:46 Pacific Standard Time
Printed: Wednesday, August 03, 2011 13:01:32 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-8-01-11.cdb 02 Aug 2011 09:02:25

Name: 110802F2_15, Date: 03-Aug-2011, Time: 01:15:51, ID: 27592_3923_046 IPR-Aqueous-4 1, Description: IPR-Aqueous-4

	# Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1 1,2,4,5,7,8-HCX	3.43e5	1.24	NO	0.220	1.000	37.25	3003.6		16.7
2	2 13C-1,2,3,7,8,9-HxCDF	1.04e6	0.51	NO	0.869	1.000	34.97	1948.2	97.4	2.31
3	3 13C-1,2,3,7,8,9-HxCDD	9.41e5	1.28	NO	0.717	1.000	34.56	2137.7	107	2.70
4	4 13C-1,2,3,4,6,9-HxCDF	1.23e6	0.52	NO	1.00	1.000	33.65	2000.0	100	2.01

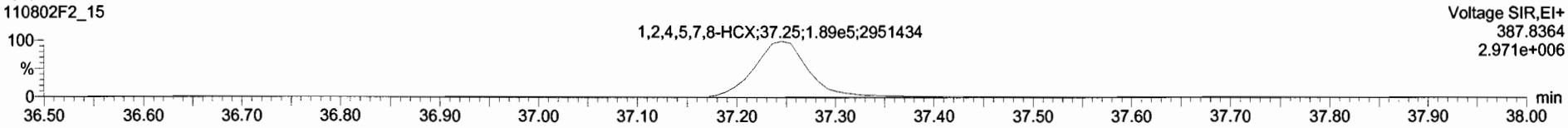
FEB 8/3/11

Dataset: Untitled

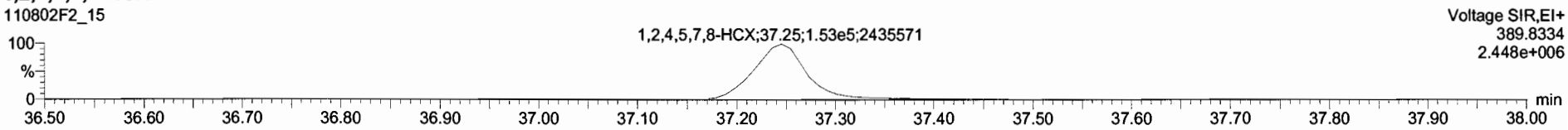
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_15, Date: 03-Aug-2011, Time: 01:15:51, ID: 27592_3923_046 IPR-Aqueous-4 1, Description: IPR-Aqueous-4

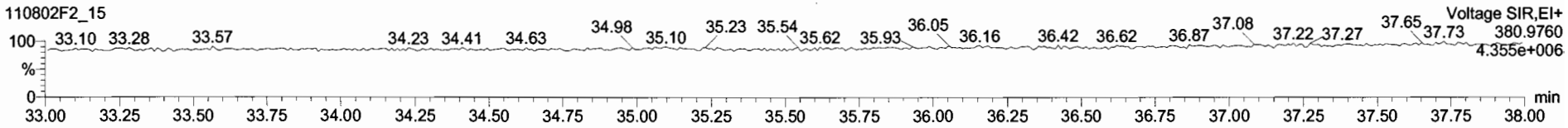
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1

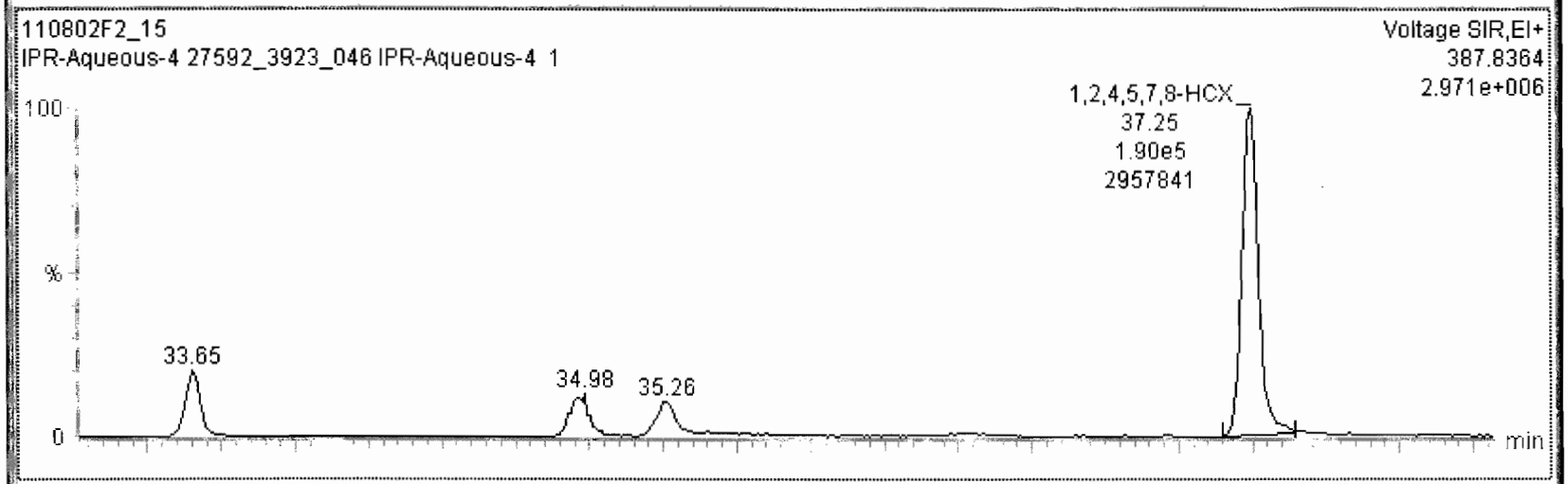


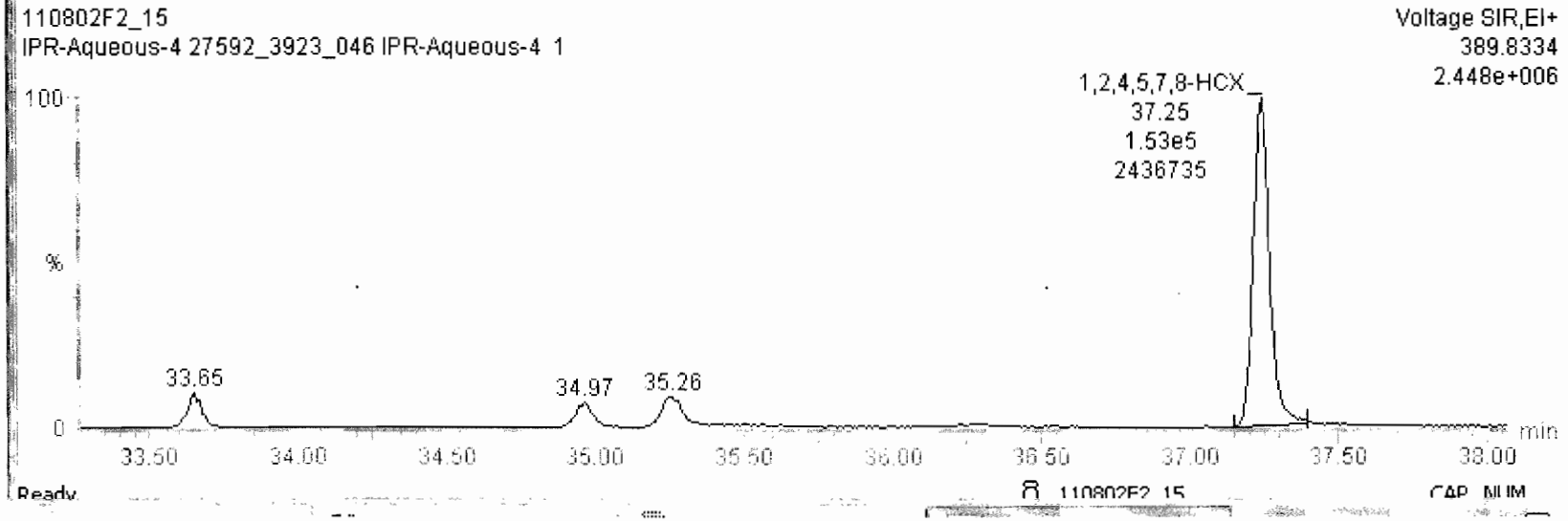
TargetLynx - 110802F2_15 - [Chromatogram]

File Edit View Display Processing Window Help

110802F2_15 - 27592_3923_046 IPR-Aqueous-4 1 - IPR-Aqueous-4

	#	Name	Resp	RA	n/y	RRF	wt/vol	RT	Conc.	%Rec	DL
1	1	1,2,4,5,7,8-HCX	3.43e5	1.24	NO	0.22	1.000	37.25	3000		16.7
2	2	13C-1,2,3,7,8,9-HxCDF	1.04e6	0.51	NO	0.87	1.000	34.97	1950	97.4	2.31
3	3	13C-1,2,3,7,8,9-HxCDD	9.41e5	1.28	NO	0.72	1.000	34.56	2140	107	2.70
4	4	13C-1,2,3,4,6,9-HxCDF	1.23e6	0.52	NO	1.00	1.000	33.65	2000	100	2.01
5	5	PFK1					1.000				



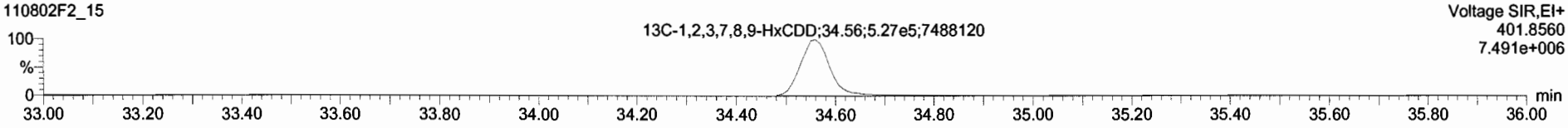


Dataset: Untitled

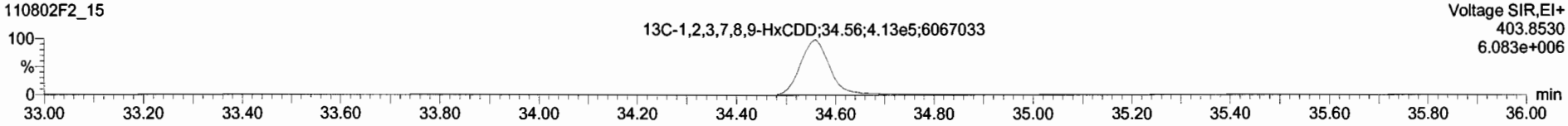
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_15, Date: 03-Aug-2011, Time: 01:15:51, ID: 27592_3923_046 IPR-Aqueous-4 1, Description: IPR-Aqueous-4

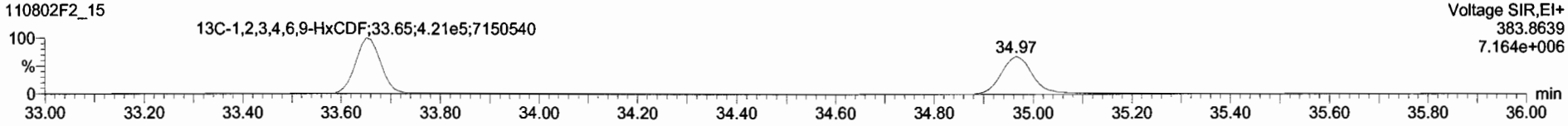
13C-1,2,3,7,8,9-HxCDD



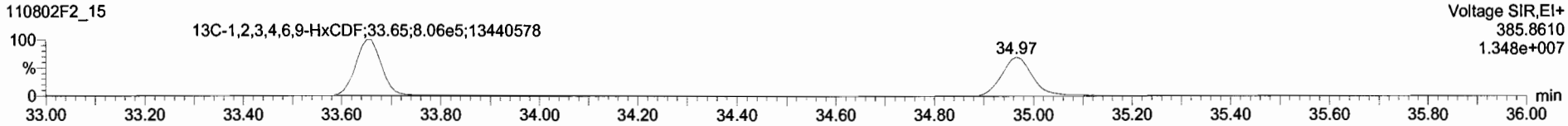
13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,4,6,9-HxCDF



13C-1,2,3,4,6,9-HxCDF



Attachment E

MDL Study Solid (Full Laboratory Deliverable)

MDL
Hexachloroxanthene

HCX

25-Jul-11

	Amt. Spiked	1	2	3	4	5	6	7	Ave	SD	MDL	RL
Hexachloroxanthene	50	45.4	42.0	41.2	40.7	47.2	44.6	36.2	42.48	3.63	11.40	20.0

Batch: 3913

Units: pg/g

PROCESS SHEET

Project No.-AR: 27592-261 of 257

Prep Due: 7/28/2011 **Project Due:** 7/14/2011

Hold Due: 7/24/2012 **TAT:** 21

Client: Vista Analytical Laboratory(AALCA01D)

Client Manager: Rose Harrelson

Method: Hexachlorophene | Hexachlorophene
Hexachloroxanthene | Hexachloroxanthene

Split Type:

Matrix: Soil

3913

LabID	Recon	Client-ID	Description	Date Received	SLoc	Shelf
008	☑	MDL-SO-1		7/25/2011	WR-2	
009	☑	MDL-SO-2		7/25/2011	WR-2	
010	☑	MDL-SO-3		7/25/2011	WR-2	
011	☑	MDL-SO-4		7/25/2011	WR-2	
012	☑	MDL-SO-5		7/25/2011	WR-2	
013	☑	MDL-SO-6		7/25/2011	WR-2	
014	☑	MDL-SO-7		7/25/2011	WR-2	

Instructions:
HCX - no split

Report Options

Report Level: TEQ Type:

EDD Type:

Report Group: Dioxins NoMDL with %Solid Samples Reconciled By: NA / /

Vial Box ID: Bacon

Date Requested 7/3/2011

Project: 27592

Extraction Set: 3913

Chemist: C. Vredeveld 7/25/11

Method(s): Hexachlorophene/Hexachloroxanthene | Hexachlorophene/Hexachloroxanthene

Prep time: 1047

C	VISTA Sample ID	G Eqv	Sample Amt. (g)	IS/NS CHEM/ WIT DATE	CRS CHEM/WIT DATE	AP CHEM/Date	~~ABSG~~ SG CHEM/Date	AA CHEM/Date	Florisil CHEM/Date	RS CHEM/WIT DATE
☐	0_3913_MB001	N/A	(10.00)	CV FEB 7/25/11	CV MC 7/27/11	N/A	CV 7/27/11	N/A	N/A	CV BB 7/27/11
☐	27592_3913_008	↓	↓	↓	↓	↓	↓	↓	↓	↓
☐	27592_3913_009									
☐	27592_3913_010									
☐	27592_3913_011									
☐	27592_3913_012									
☐	27592_3913_013									
☐	27592_3913_014									

IS Name

NS Name MDL

CRS Name

RS Name

PCDD/F | PCDD/F | PCDD/F | PCDD/F

PCB | PCB 110713A 25µL | PCB | PCB

HCX 110706A 10µL | HCX ~~HG701A 10µL~~ CV 7/25 | HCX 110503B 10µL | HCX 110503C 10µL

Cycle Time 7/25
Start: 1610
Stop: 0825 7/26

APP.: SEFUN (SOX) SDS

SOLV: DCM

Other: N/A

Final Volume(s): 20µL CV

Check Out:
Chemist: NA / /

Check-In:
Chemist: NA / /

Balance ID: NA

Comments:

hrmsgenprepL.rpt

CALIBRATION STANDARDS REVIEW CHECKLIST

Vista Analytical Laboratory

Beg. Calibration ID: ST110727F3-4

End Calibration ID: N/A

	Beg.	End
Ion abundance within QC limits?	✓	N/A
Concentration within range?	✓	
First and last eluters present?	N/A	
Retention Times within criteria?	✓	
Verification Std. named correctly? (ST-Year-Month-Day-VG ID)	✓	
Forms signed and dated?	✓	
Correct ICAL referenced?	✓	
Run Log:		
-Data file matches Conc Cal ID?	✓	
-Correct instrument listed?	✓	
-Samples within 12-hour clock?	Y	n

	Beg.	End
Mass resolution > 10,000? ▪ Method 1614 > 5,000; CARB 429 > 8,000	✓	✓
TCDD/TCDF valleys < 25%?	N/A	N/A
Peaks integrated correctly?	✓	
Manual integrations included?	✓	
8280 CS1 Ending Standard		
-Ratios within limits		
-S/N > 2.5:1		
-CS1 within 12-hour clock		✓

Comments:

Reviewed by: DMS 7/28/11
Initials & Date

* *Ending standard criteria applicable to 8290 only.*

Quantify Compound Summary Report **MassLynx 4.1**
Vista Analytical Laboratory VG-9

Dataset: Untitled

Last Altered: Thursday, July 28, 2011 09:28:30 Pacific Standard Time
Printed: Thursday, July 28, 2011 09:28:40 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-7-27-11.cdb 28 Jul 2011 08:40:50

Compound name: 1,2,4,5,7,8-HCX

	Name	ID	Acq.Date	Acq.Time
1	110727F3_1	ST110727F3-1 HCX CS0 110706B	27-Jul-11	15:55:03
2	110727F3_2	ST110727F3-2 HCX CS1 110706C	27-Jul-11	16:37:07
3	110727F3_3	ST110727F3-3 HCX CS2 110706D	27-Jul-11	17:20:45
4	110727F3_4	ST110727F3-4 HCX CS3 110706E	27-Jul-11	18:04:22
5	110727F3_5	ST110727F3-5 HCX CS4 110706F	27-Jul-11	18:48:00
6	110727F3_6	ST110727F3-6 HCX CS5 110706G	27-Jul-11	19:31:38
7	110727F3_7	ST110727F3-7 HCX CS6 110706H	27-Jul-11	20:15:10
8	110727F3_8	SOLVENT BLANK	27-Jul-11	20:58:48
9	110727F3_9	SOLVENT BLANK	27-Jul-11	21:42:26
10	110727F3_10	0_3913_MB001	27-Jul-11	22:26:04
11	110727F3_11	27592_3913_008 MDL-SO-1 10	27-Jul-11	23:09:40
12	110727F3_12	27592_3913_009 MDL-SO-2 10	27-Jul-11	23:53:12
13	110727F3_13	27592_3913_010 MDL-SO-3 10	28-Jul-11	00:36:50
14	110727F3_14	27592_3913_011 MDL-SO-4 10	28-Jul-11	01:20:21
15	110727F3_15	27592_3913_012 MDL-SO-5 10	28-Jul-11	02:03:59
16	110727F3_16	27592_3913_013 MDL-SO-6 10	28-Jul-11	02:47:39
17	110727F3_17	27592_3913_014 MDL-SO-7 10	28-Jul-11	03:31:17
18	110727F3_18	SOLVENT BLANK	28-Jul-11	04:14:50

Dataset: Untitled

Last Altered: Thursday, July 28, 2011 9:38:53 AM Pacific Standard Time
Printed: Thursday, July 28, 2011 9:40:35 AM Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-7-27-11.cdb 28 Jul 2011 08:40:50

Name: 110727F3_10, Date: 27-Jul-2011, Time: 22:26:04, ID: 0_3913_MB001, Description: 0_3913_MB001

	# Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1 1,2,4,5,7,8-HCX	3.34e3	1.39	NO	0.210	10.000	37.26	1.2780		1.64
2	2 13C-1,2,3,7,8,9-HxCDF	2.50e6	0.52	NO	0.874	10.000	34.98	188.93	94.5	0.199
3	3 13C-1,2,3,7,8,9-HxCDD	2.30e6	1.17	NO	0.799	10.000	34.57	190.52	95.3	0.147
4	4 13C-1,2,3,4,6,9-HxCDF	3.02e6	0.54	NO	1.00	10.000	33.66	200.00	100	0.174

RL=20

FEB 7/28/11

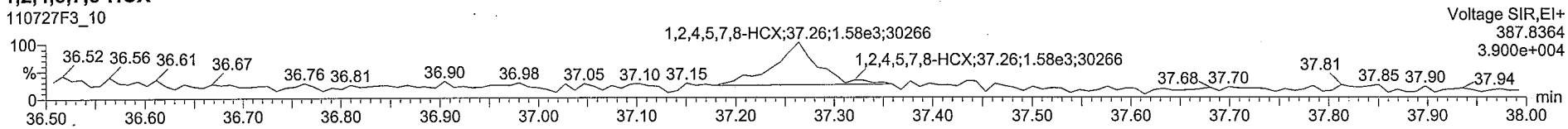
Dataset: Untitled

Last Altered: Thursday, July 28, 2011 09:26:41 Pacific Standard Time
Printed: Thursday, July 28, 2011 09:27:31 Pacific Standard Time

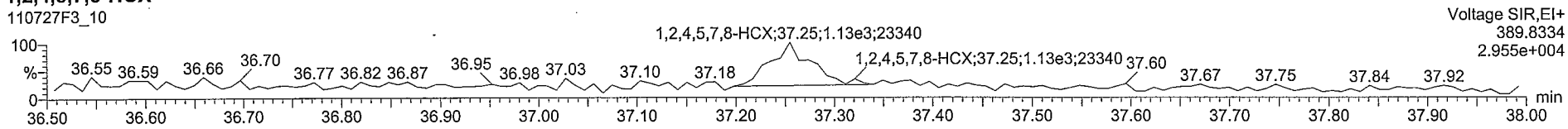
Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-7-27-11.cdb 28 Jul 2011 08:40:50

Name: 110727F3_10, Date: 27-Jul-2011, Time: 22:26:04, ID: 0_3913_MB001, Description: 0_3913_MB001

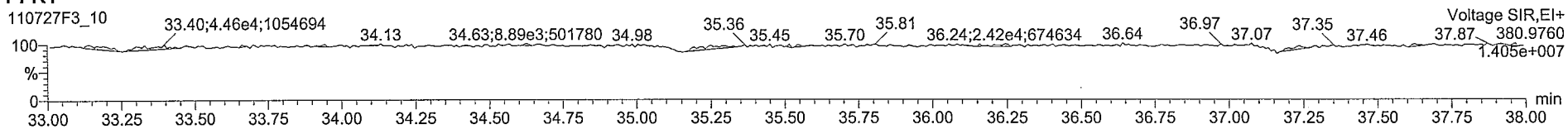
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1



TargetLynx - untitled * - [Chromatogram]

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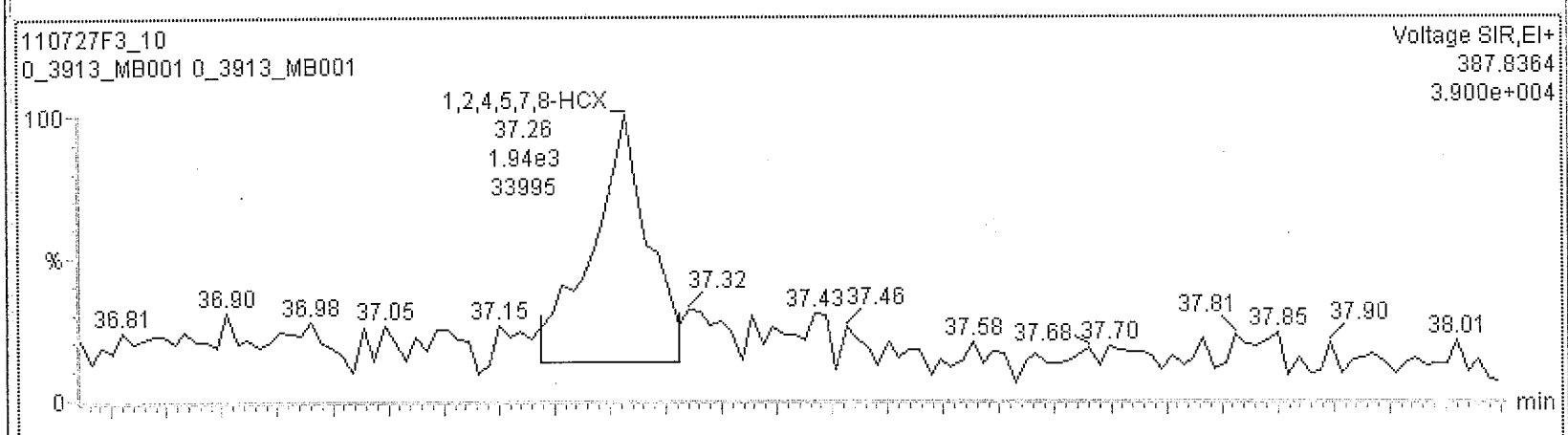
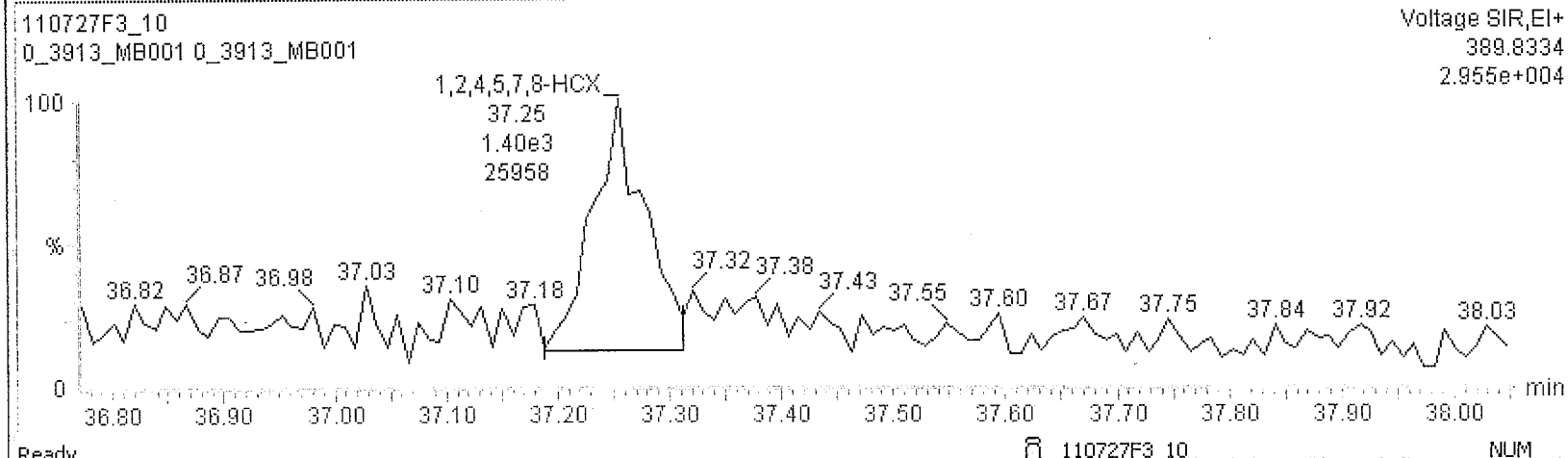
110727F3_10 0_3913_MB001 0_3913_MB001

	#	Name	Resp	RA	n/y	RRF	wt/vol	RT	Conc.	%Rec	DL
1	1	1,2,4,5,7,8-HCX	3.34e3	1.39	NO	0.21	10.000	37.26	1.28		1.64
2	2	13C-1,2,3,7,8,9-HxCDF	2.50e6	0.52	NO	0.87	10.000	34.98	189	94.5	0.199
3	3	13C-1,2,3,7,8,9-HxCDD	2.30e6	1.17	NO	0.80	10.000	34.57	191	95.3	0.147
4	4	13C-1,2,3,4,6,9-HxCDF	3.02e6	0.54	NO	1.00	10.000	33.66	200	100	0.174
5	5	PFK1					1.000				





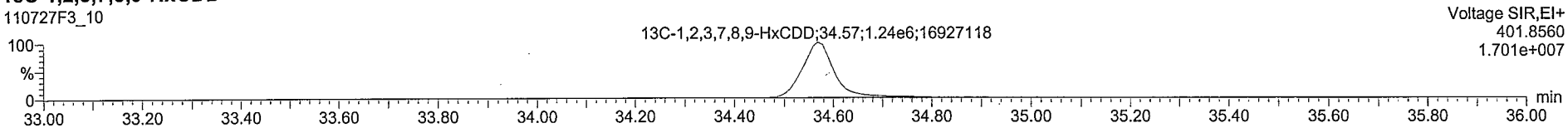
Ready 110727F3 10 NUM

Dataset: Untitled

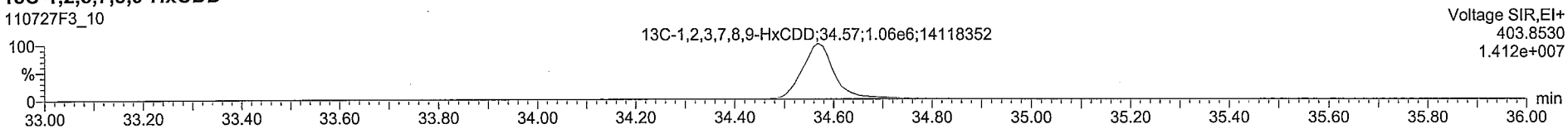
Last Altered: Thursday, July 28, 2011 09:26:41 Pacific Standard Time
Printed: Thursday, July 28, 2011 09:27:31 Pacific Standard Time

Name: 110727F3_10, Date: 27-Jul-2011, Time: 22:26:04, ID: 0_3913_MB001, Description: 0_3913_MB001

13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,7,8,9-HxCDD



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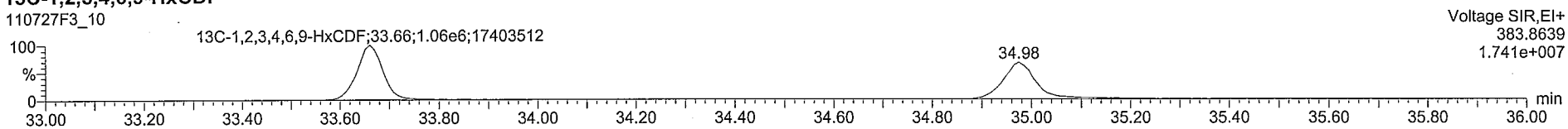
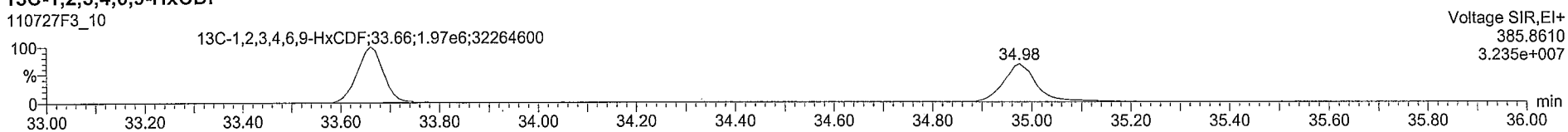


13C-1,2,3,4,6,9-HxCDF



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Last Altered: Thursday, July 28, 2011 11:27:43 Pacific Standard Time
Printed: Thursday, July 28, 2011 11:28:04 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-7-27-11.cdb 28 Jul 2011 08:40:50

Name: 110727F3_11, Date: 27-Jul-2011, Time: 23:09:40, ID: 27592_3913_008 MDL-SO-1 10, Description: MDL-SO-1

	# Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1 1,2,4,5,7,8-HCX	1.24e5	1.22	NO	0.210	10.000	37.26	45.397		1.19
2	2 13C-1,2,3,7,8,9-HxCDF	2.61e6	0.54	NO	0.874	10.000	34.98	196.95	98.5	0.172
3	3 13C-1,2,3,7,8,9-HxCDD	2.49e6	1.22	NO	0.799	10.000	34.58	205.40	103	0.155
4	4 13C-1,2,3,4,6,9-HxCDF	3.03e6	0.54	NO	1.00	10.000	33.66	200.00	100	0.150

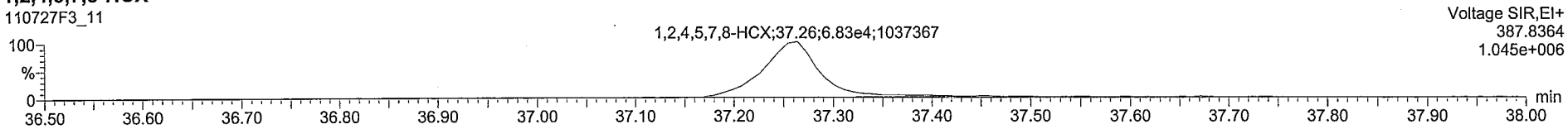
FEB 7/28/11

Dataset: Untitled

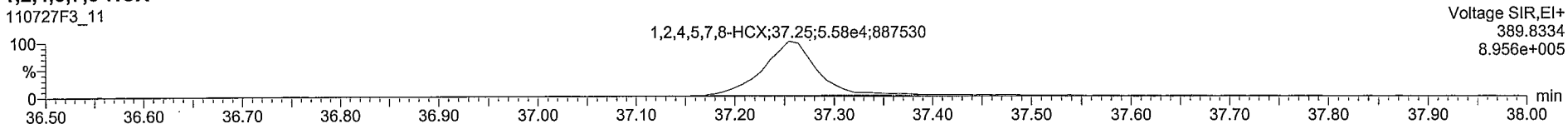
Last Altered: Thursday, July 28, 2011 09:26:41 Pacific Standard Time
Printed: Thursday, July 28, 2011 09:27:31 Pacific Standard Time

Name: 110727F3_11, Date: 27-Jul-2011, Time: 23:09:40, ID: 27592_3913_008 MDL-SO-1 10, Description: MDL-SO-1

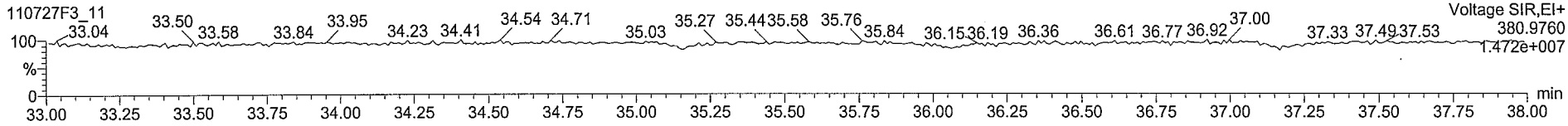
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1

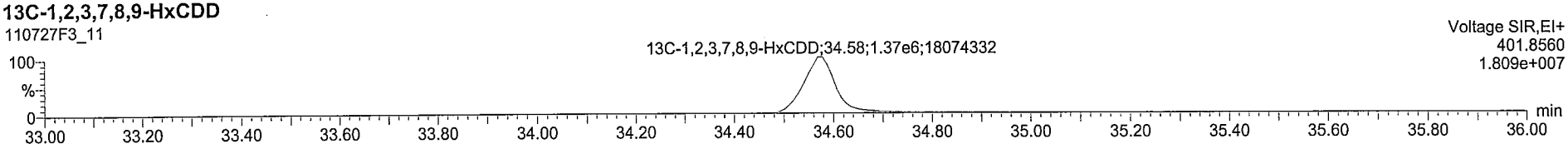


Dataset: Untitled

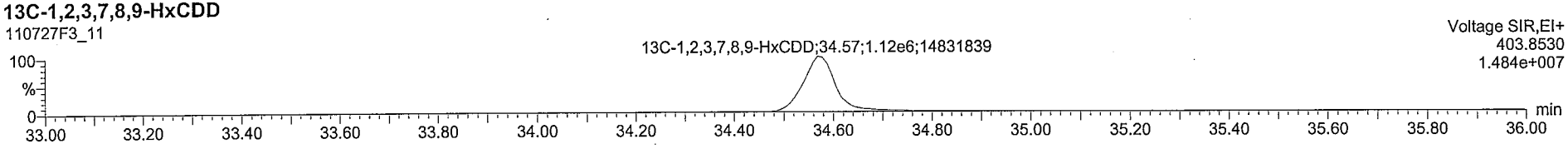
Last Altered: Thursday, July 28, 2011 09:26:41 Pacific Standard Time
Printed: Thursday, July 28, 2011 09:27:31 Pacific Standard Time

Name: 110727F3_11, Date: 27-Jul-2011, Time: 23:09:40, ID: 27592_3913_008 MDL-SO-1 10, Description: MDL-SO-1

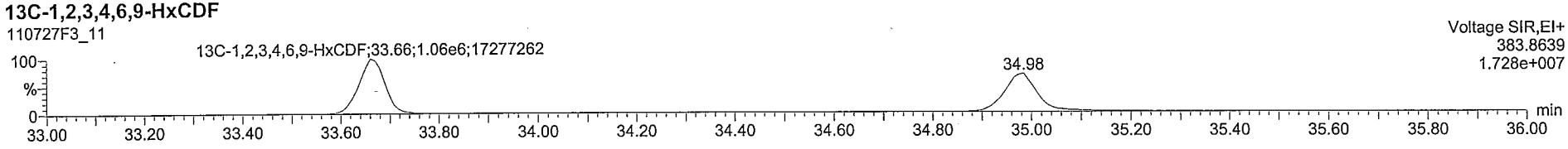
13C-1,2,3,7,8,9-HxCDD



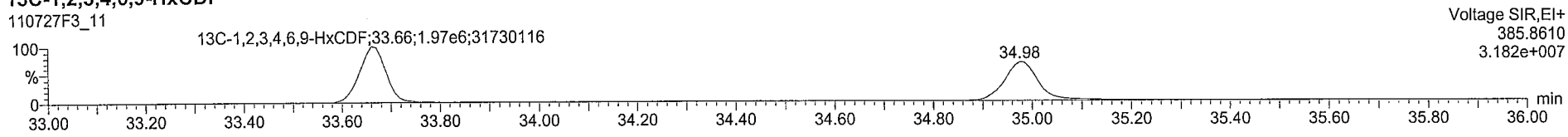
13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,4,6,9-HxCDF



13C-1,2,3,4,6,9-HxCDF



Dataset: C:\MassLynx\Default.pro\Results\110727F3\110727F3-12.qld

Last Altered: Thursday, July 28, 2011 11:31:13 Pacific Standard Time
Printed: Thursday, July 28, 2011 11:32:36 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-7-27-11.cdb 28 Jul 2011 08:40:50

Name: 110727F3_12, Date: 27-Jul-2011, Time: 23:53:12, ID: 27592_3913_009 MDL-SO-2 10, Description: MDL-SO-2

	# Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1 1,2,4,5,7,8-HCX	1.17e5	1.23	NO	0.210	10.000	37.26	42.040		1.22
2	2 13C-1,2,3,7,8,9-HxCDF	2.66e6	0.50	NO	0.874	10.000	34.99	204.97	102	0.177
3	3 13C-1,2,3,7,8,9-HxCDD	2.43e6	1.21	NO	0.799	10.000	34.58	204.66	102	0.197
4	4 13C-1,2,3,4,6,9-HxCDF	2.97e6	0.53	NO	1.00	10.000	33.67	200.00	100	0.155

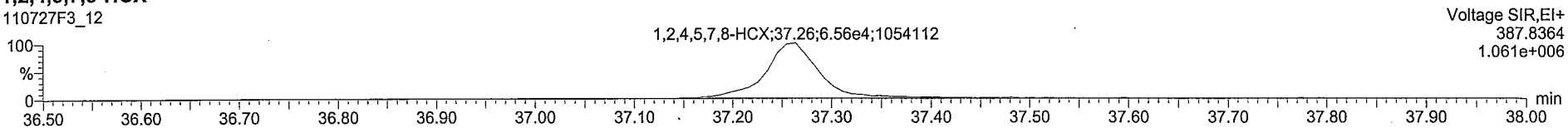
FEB 7/28/11

Dataset: Untitled

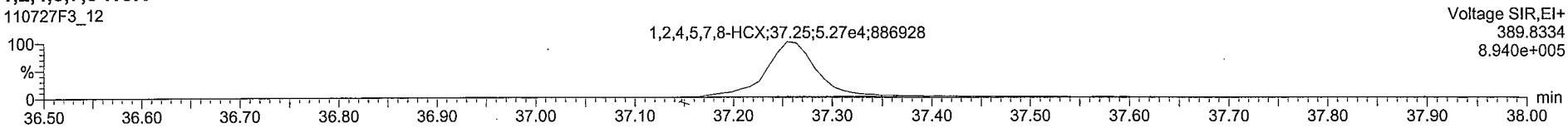
Last Altered: Thursday, July 28, 2011 09:26:41 Pacific Standard Time
Printed: Thursday, July 28, 2011 09:27:31 Pacific Standard Time

Name: 110727F3_12, Date: 27-Jul-2011, Time: 23:53:12, ID: 27592_3913_009 MDL-SO-2 10, Description: MDL-SO-2

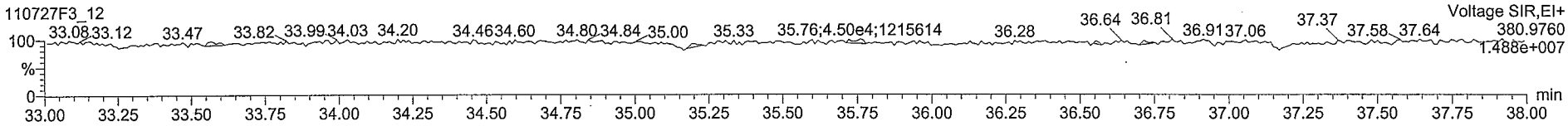
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1

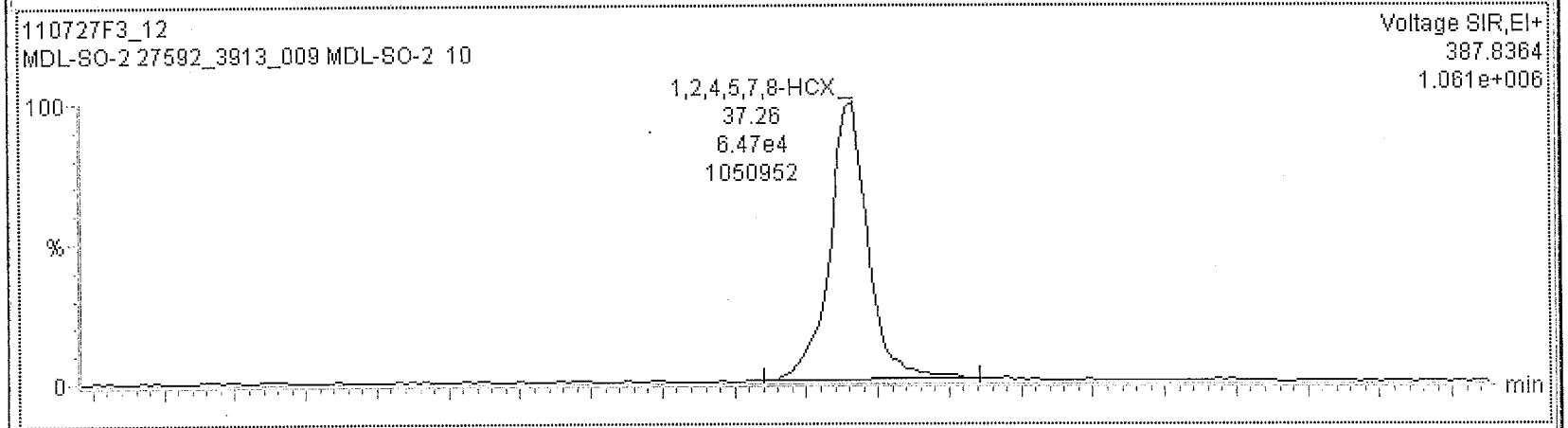


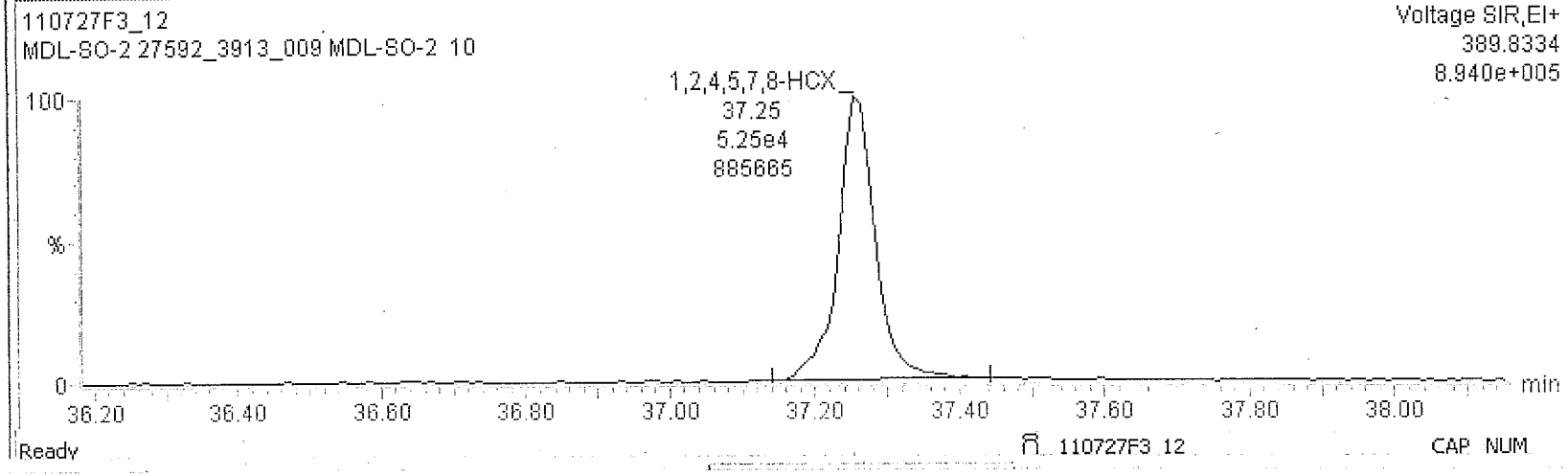
TargetLynx - untitled * - [Chromatogram]

File Edit View Display Processing Window Help

110727F3_12 27592_3913_009 MDL-SO-2 10 MDL-SO-2

	#	Name	Resp	RA	n/y	RRF	wt/vol	RT	Conc.	%Rec	DL
1	1	1,2,4,5,7,8-HCX	1.17e5	1.23	NO	0.21	10.000	37.26	42.0		1.22
2	2	13C-1,2,3,7,8,9-HxCDF	2.66e6	0.50	NO	0.87	10.000	34.98	205	102	0.177
3	3	13C-1,2,3,7,8,9-HxCDD	2.43e6	1.21	NO	0.80	10.000	34.58	205	102	0.197
4	4	13C-1,2,3,4,6,9-HxCDF	2.97e6	0.53	NO	1.00	10.000	33.67	200	100	0.155
5	5	PFK1					1.000				





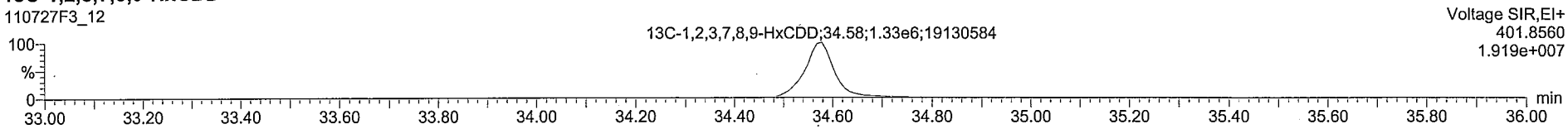
Ready 110727F3 12 CAP NUM

Dataset: Untitled

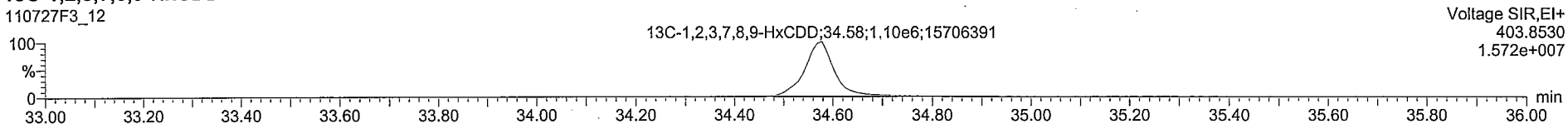
Last Altered: Thursday, July 28, 2011 09:26:41 Pacific Standard Time
Printed: Thursday, July 28, 2011 09:27:31 Pacific Standard Time

Name: 110727F3_12, Date: 27-Jul-2011, Time: 23:53:12, ID: 27592_3913_009 MDL-SO-2 10, Description: MDL-SO-2

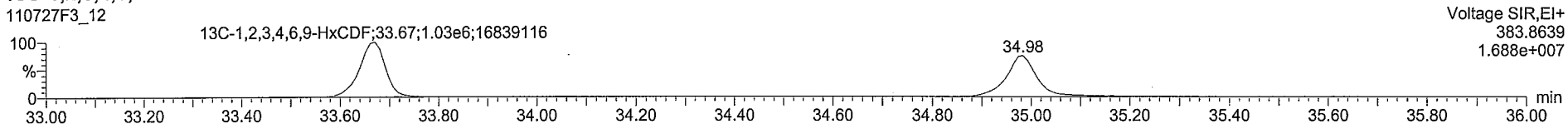
13C-1,2,3,7,8,9-HxCDD



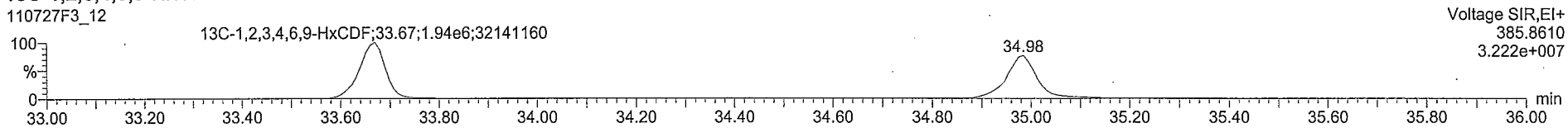
13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,4,6,9-HxCDF



13C-1,2,3,4,6,9-HxCDF



Dataset: C:\MassLynx\Default.pro\Results\110727F3\110727F3-13.qld

Last Altered: Thursday, July 28, 2011 11:33:07 Pacific Standard Time
Printed: Thursday, July 28, 2011 11:34:08 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-7-27-11.cdb 28 Jul 2011 08:40:50

Name: 110727F3_13, Date: 28-Jul-2011, Time: 00:36:50, ID: 27592_3913_010 MDL-SO-3 10, Description: MDL-SO-3

	# Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1 1,2,4,5,7,8-HCX	1.22e5	1.27	NO	0.210	10.000	37.26	41.220		0.612
2	2 13C-1,2,3,7,8,9-HxCDF	2.83e6	0.51	NO	0.874	10.000	34.98	211.61	106	0.179
3	3 13C-1,2,3,7,8,9-HxCDD	2.48e6	1.21	NO	0.799	10.000	34.58	202.68	101	0.142
4	4 13C-1,2,3,4,6,9-HxCDF	3.06e6	0.51	NO	1.00	10.000	33.67	200.00	100	0.156

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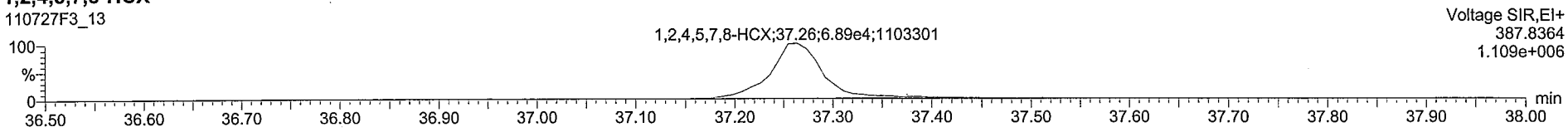
Dataset: Untitled

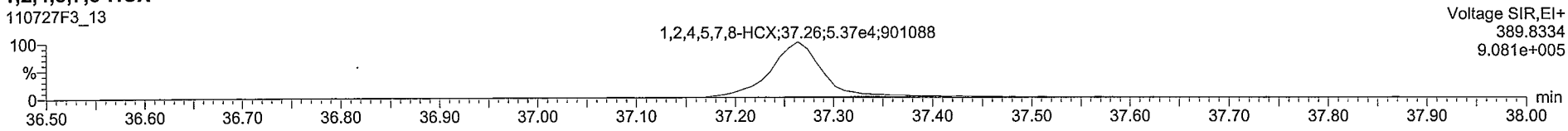
Last Altered: Thursday, July 28, 2011 09:26:41 Pacific Standard Time
Printed: Thursday, July 28, 2011 09:27:31 Pacific Standard Time

Name: 110727F3_13, Date: 28-Jul-2011, Time: 00:36:50, ID: 27592_3913_010 MDL-SO-3 10, Description: MDL-SO-3

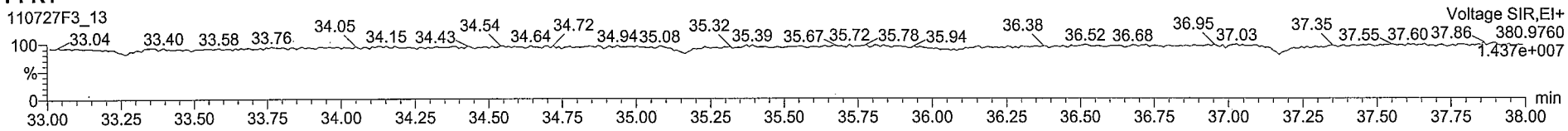
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1



TargetLynx - untitled * - [Chromatogram]

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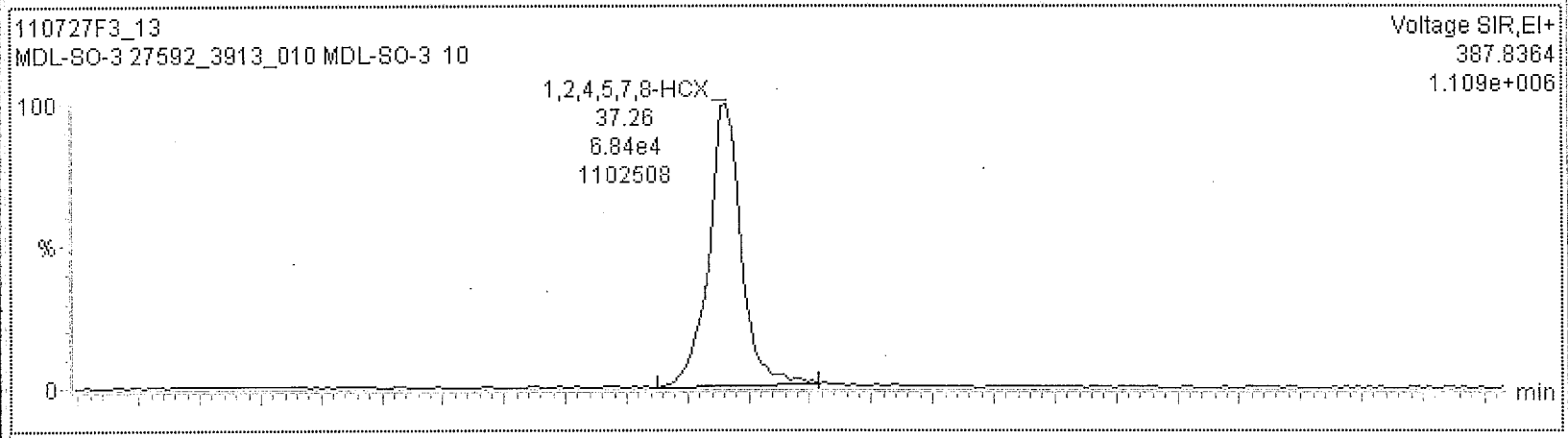
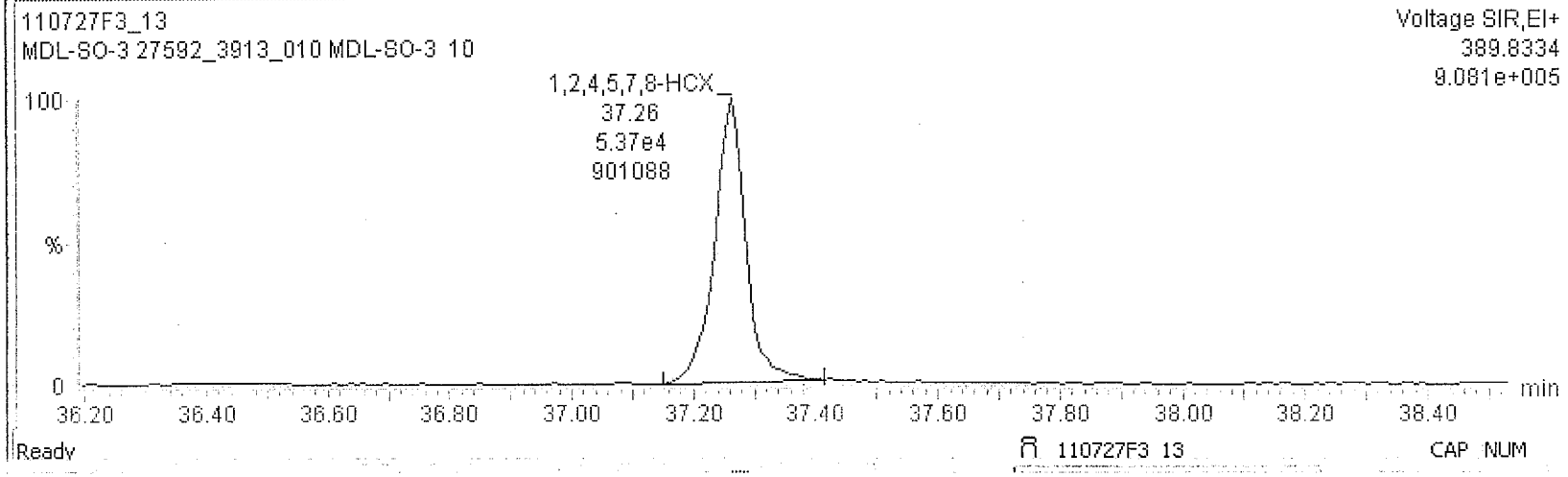
110727F3_13 27592_3913_010 MDL-SO-3 10 MDL-SO-3

	#	Name	Resp	RA	n/y	RRF	wt/vol	RT	Conc.	%Rec	DL
1	1	1,2,4,5,7,8-HCX	1.22e5	1.27	NO	0.21	10.000	37.26	41.2		0.612
2	2	13C-1,2,3,7,8,9-HxCDF	2.83e6	0.51	NO	0.87	10.000	34.98	212	106	0.179
3	3	13C-1,2,3,7,8,9-HxCDD	2.48e6	1.21	NO	0.80	10.000	34.58	203	101	0.142
4	4	13C-1,2,3,4,6,9-HxCDF	3.06e6	0.51	NO	1.00	10.000	33.67	200	100	0.156
5	5	PFK1					1.000				





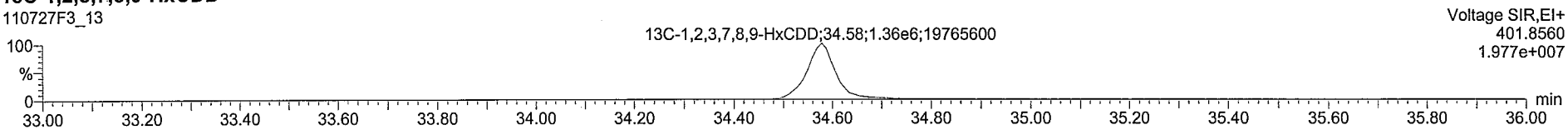
Ready 110727F3 13 CAP NUM

Dataset: Untitled

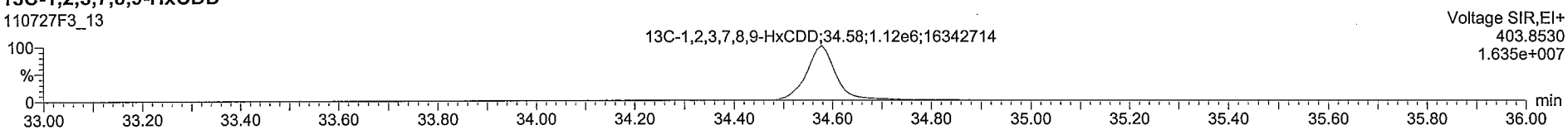
Last Altered: Thursday, July 28, 2011 09:26:41 Pacific Standard Time
Printed: Thursday, July 28, 2011 09:27:31 Pacific Standard Time

Name: 110727F3_13, Date: 28-Jul-2011, Time: 00:36:50, ID: 27592_3913_010 MDL-SO-3 10, Description: MDL-SO-3

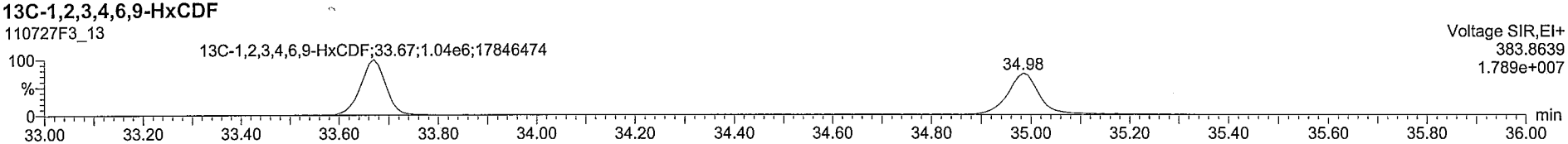
13C-1,2,3,7,8,9-HxCDD



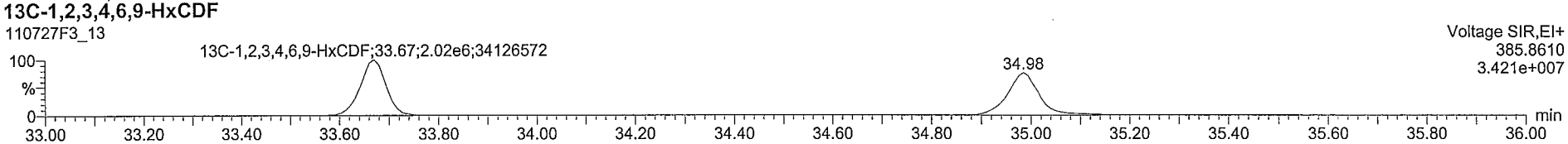
13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,4,6,9-HxCDF



13C-1,2,3,4,6,9-HxCDF



Dataset: C:\MassLynx\Default.pro\Results\110727F3\110727F3-14.qld

Last Altered: Thursday, July 28, 2011 11:35:48 Pacific Standard Time
Printed: Thursday, July 28, 2011 11:36:24 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-7-27-11.cdb 28 Jul 2011 08:40:50

Name: 110727F3_14, Date: 28-Jul-2011, Time: 01:20:21, ID: 27592_3913_011 MDL-SO-4 10, Description: MDL-SO-4

	# Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1 1,2,4,5,7,8-HCX	1.18e5	1.19	NO	0.210	10.000	37.27	40.740		0.479
2	2 13C-1,2,3,7,8,9-HxCDF	2.76e6	0.53	NO	0.874	10.000	34.98	204.93	102	0.161
3	3 13C-1,2,3,7,8,9-HxCDD	2.63e6	1.22	NO	0.799	10.000	34.59	213.45	107	0.137
4	4 13C-1,2,3,4,6,9-HxCDF	3.08e6	0.53	NO	1.00	10.000	33.68	200.00	100	0.140

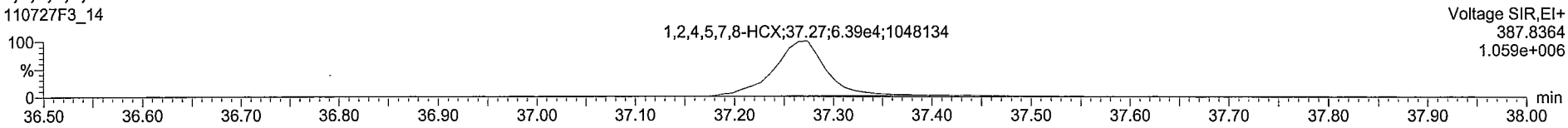
FEB 7/28/11

Dataset: Untitled

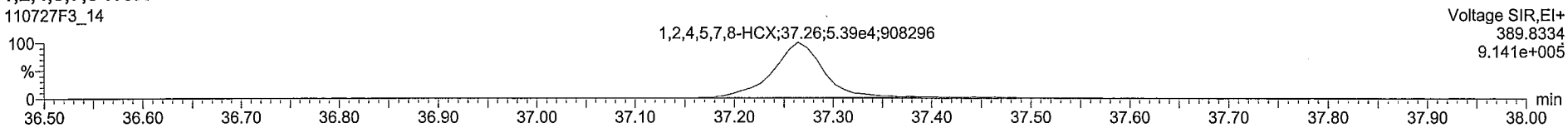
Last Altered: Thursday, July 28, 2011 09:26:41 Pacific Standard Time
Printed: Thursday, July 28, 2011 09:27:31 Pacific Standard Time

Name: 110727F3_14, Date: 28-Jul-2011, Time: 01:20:21, ID: 27592_3913_011 MDL-SO-4 10, Description: MDL-SO-4

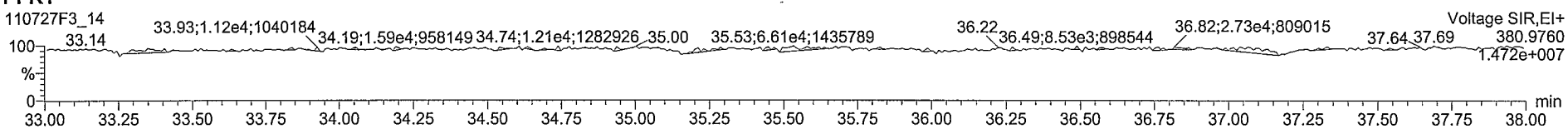
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1

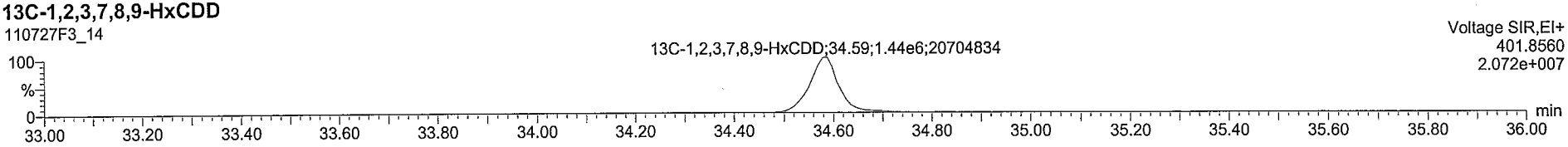


Dataset: Untitled

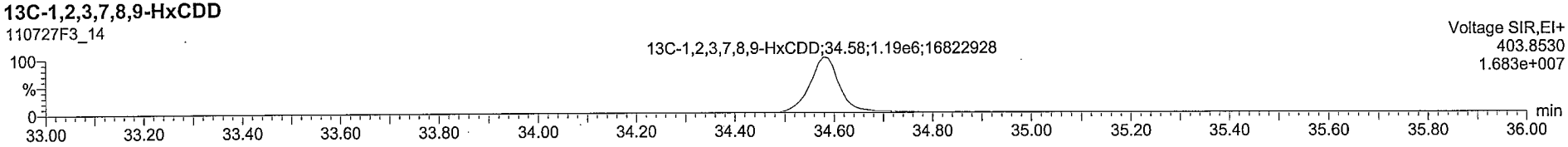
Last Altered: Thursday, July 28, 2011 09:26:41 Pacific Standard Time
Printed: Thursday, July 28, 2011 09:27:31 Pacific Standard Time

Name: 110727F3_14, Date: 28-Jul-2011, Time: 01:20:21, ID: 27592_3913_011 MDL-SO-4 10, Description: MDL-SO-4

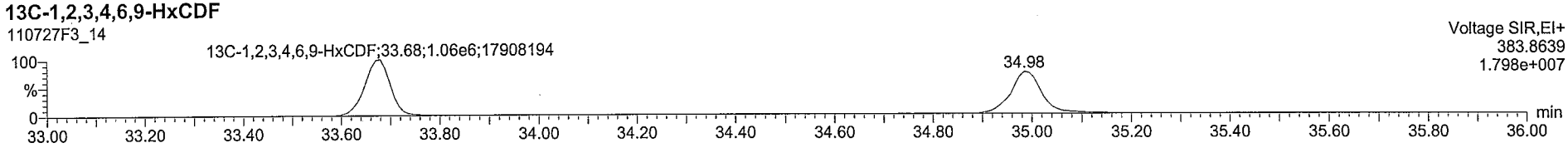
13C-1,2,3,7,8,9-HxCDD



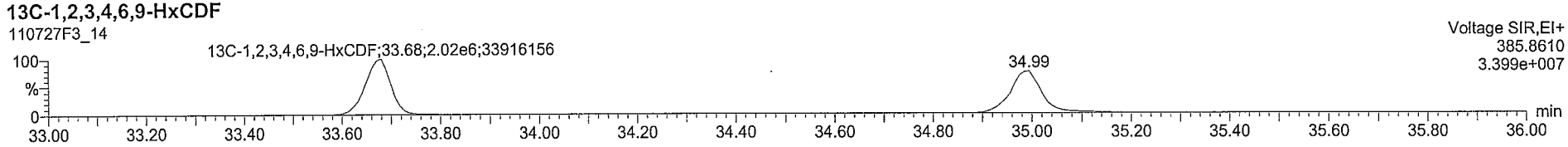
13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,4,6,9-HxCDF



13C-1,2,3,4,6,9-HxCDF



Dataset: C:\MassLynx\Default.pro\Results\110727F3\110727F3-15.qld

Last Altered: Thursday, July 28, 2011 11:37:38 Pacific Standard Time
Printed: Thursday, July 28, 2011 11:38:53 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-7-27-11.cdb 28 Jul 2011 08:40:50

Name: 110727F3_15, Date: 28-Jul-2011, Time: 02:03:59, ID: 27592_3913_012 MDL-SO-5 10, Description: MDL-SO-5

	# Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1 1,2,4,5,7,8-HCX	1.27e5	1.28	NO	0.210	10.000	37.25	47.156		2.91
2	2 13C-1,2,3,7,8,9-HxCDF	2.58e6	0.53	NO	0.874	10.000	34.97	203.46	102	0.159
3	3 13C-1,2,3,7,8,9-HxCDD	2.36e6	1.18	NO	0.799	10.000	34.57	203.46	102	0.138
4	4 13C-1,2,3,4,6,9-HxCDF	2.90e6	0.52	NO	1.00	10.000	33.66	200.00	100	0.139

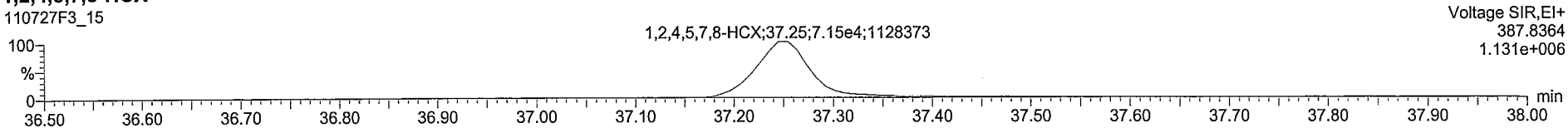
FEB 7/28/11

Dataset: Untitled

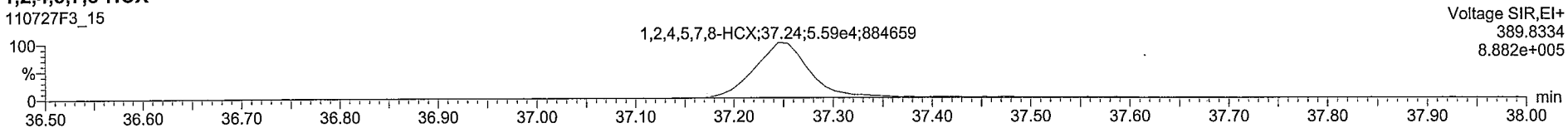
Last Altered: Thursday, July 28, 2011 09:26:41 Pacific Standard Time
Printed: Thursday, July 28, 2011 09:27:31 Pacific Standard Time

Name: 110727F3_15, Date: 28-Jul-2011, Time: 02:03:59, ID: 27592_3913_012 MDL-SO-5 10, Description: MDL-SO-5

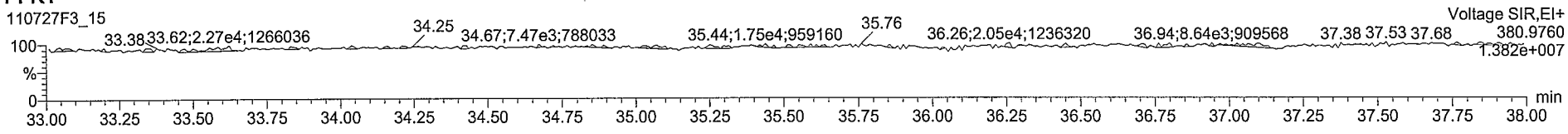
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1

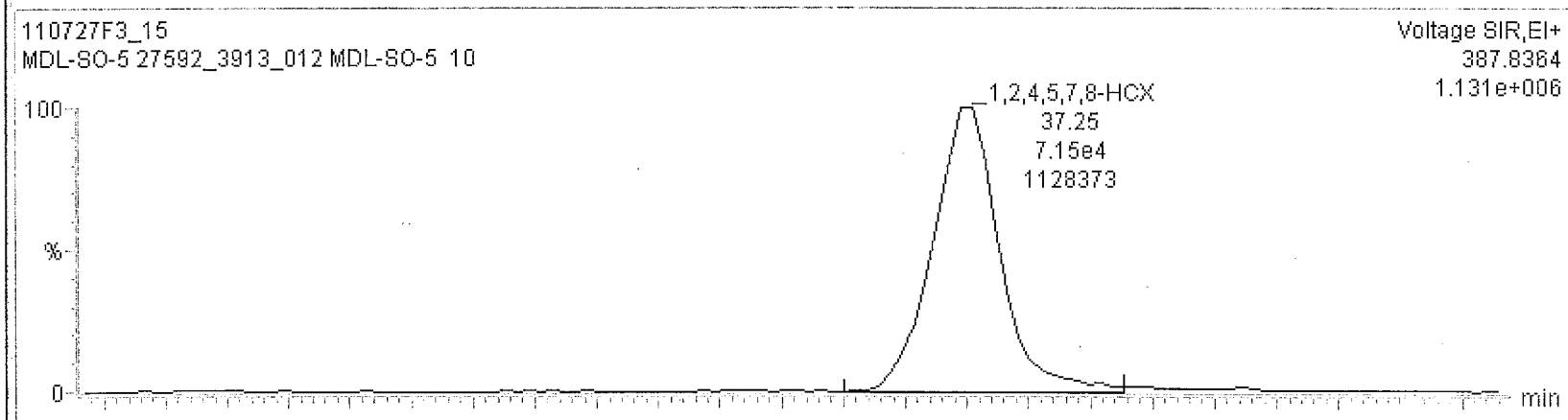


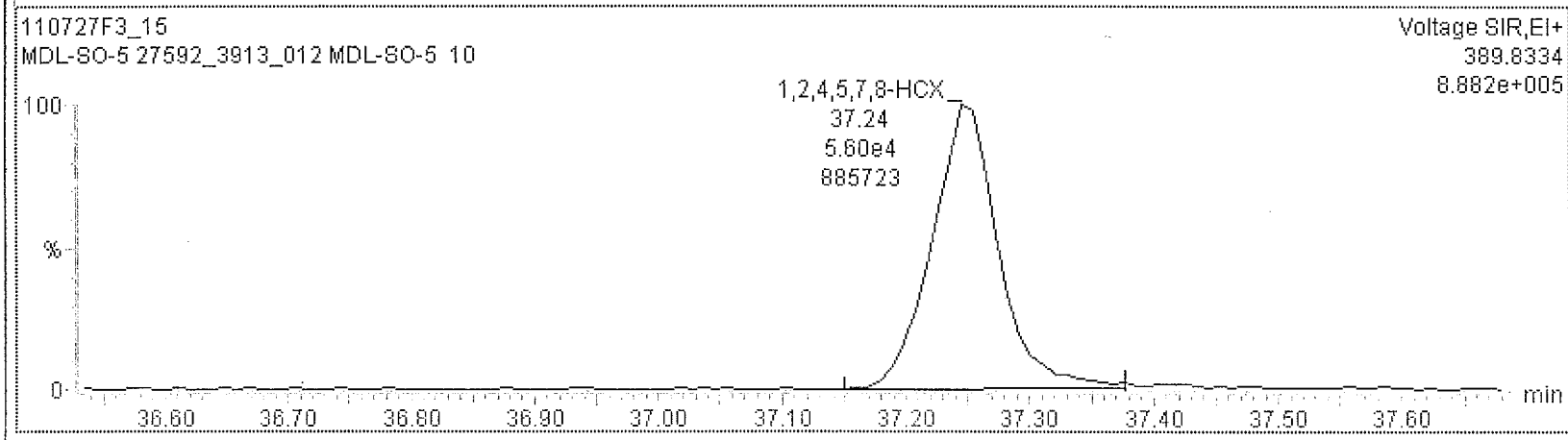
TargetLynx - untitled * - [Chromatogram]

File Edit View Display Processing Window Help

110727F3_15 27592_3913_012 MDL-SO-5 10 MDL-SO-5

	#	Name	Resp	RA	n/y	RRF	wt/vol	RT	Conc.	%Rec	DL
1	1	1,2,4,5,7,8-HCX	1.27e5	1.28	NO	0.21	10.000	37.25	47.2		2.91
2	2	13C-1,2,3,7,8,9-HxCDF	2.58e6	0.53	NO	0.87	10.000	34.97	203	102	0.159
3	3	13C-1,2,3,7,8,9-HxCDD	2.36e6	1.18	NO	0.80	10.000	34.57	203	102	0.138
4	4	13C-1,2,3,4,6,9-HxCDF	2.90e6	0.52	NO	1.00	10.000	33.66	200	100	0.139
5	5	PFK1					1.000				





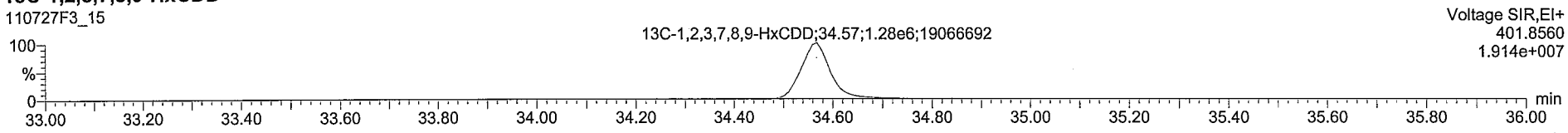
Ready 110727F3 15 CAP NUM

Dataset: Untitled

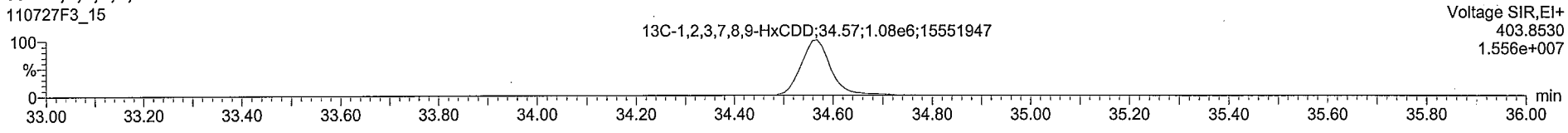
Last Altered: Thursday, July 28, 2011 09:26:41 Pacific Standard Time
Printed: Thursday, July 28, 2011 09:27:31 Pacific Standard Time

Name: 110727F3_15, Date: 28-Jul-2011, Time: 02:03:59, ID: 27592_3913_012 MDL-SO-5 10, Description: MDL-SO-5

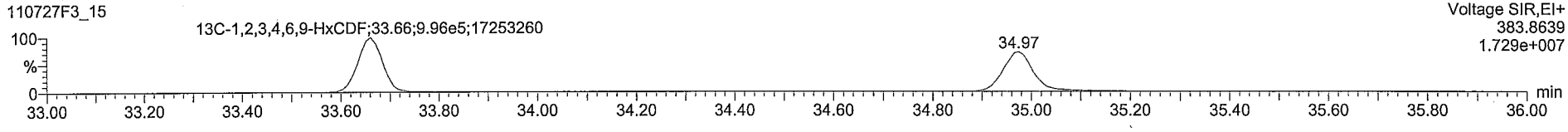
13C-1,2,3,7,8,9-HxCDD



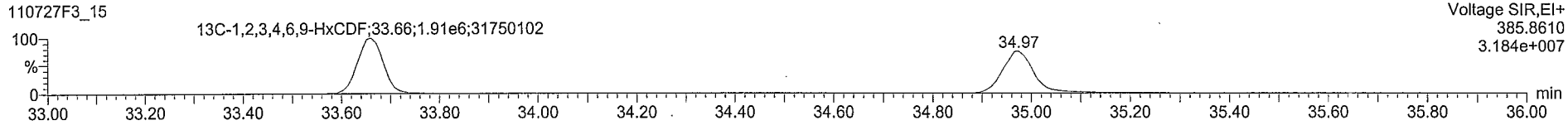
13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,4,6,9-HxCDF



13C-1,2,3,4,6,9-HxCDF



Dataset: C:\MassLynx\Default.pro\Results\110727F3\110727F3-16.qld

Last Altered: Thursday, July 28, 2011 11:40:38 Pacific Standard Time
Printed: Thursday, July 28, 2011 11:41:08 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-7-27-11.cdb 28 Jul 2011 08:40:50

Name: 110727F3_16, Date: 28-Jul-2011, Time: 02:47:39, ID: 27592_3913_013 MDL-SO-6 10, Description: MDL-SO-6

	# Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1 1,2,4,5,7,8-HCX	1.20e5	1.27	NO	0.210	10.000	37.25	44.577		0.495
2	2 13C-1,2,3,7,8,9-HxCDF	2.56e6	0.53	NO	0.874	10.000	34.98	202.39	101	0.155
3	3 13C-1,2,3,7,8,9-HxCDD	2.35e6	1.19	NO	0.799	10.000	34.57	203.15	102	0.149
4	4 13C-1,2,3,4,6,9-HxCDF	2.90e6	0.53	NO	1.00	10.000	33.66	200.00	100	0.135

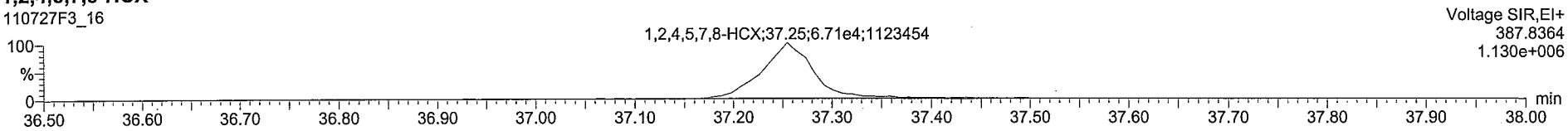
FEB 7/28/11

Dataset: Untitled

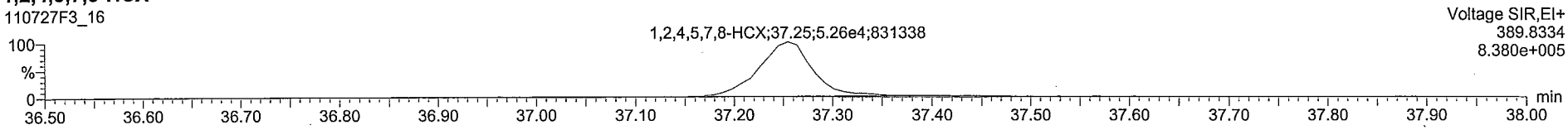
Last Altered: Thursday, July 28, 2011 09:26:41 Pacific Standard Time
Printed: Thursday, July 28, 2011 09:27:31 Pacific Standard Time

Name: 110727F3_16, Date: 28-Jul-2011, Time: 02:47:39, ID: 27592_3913_013 MDL-SO-6 10, Description: MDL-SO-6

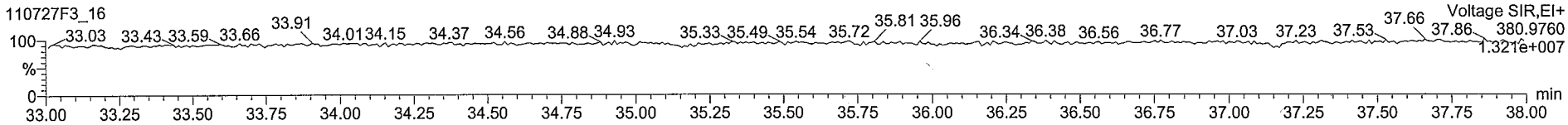
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1

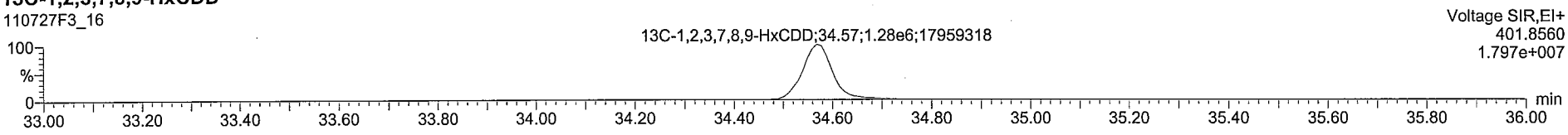


Dataset: Untitled

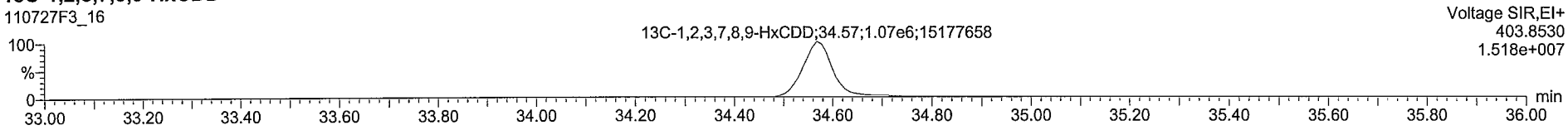
Last Altered: Thursday, July 28, 2011 09:26:41 Pacific Standard Time
Printed: Thursday, July 28, 2011 09:27:31 Pacific Standard Time

Name: 110727F3_16, Date: 28-Jul-2011, Time: 02:47:39, ID: 27592_3913_013 MDL-SO-6 10, Description: MDL-SO-6

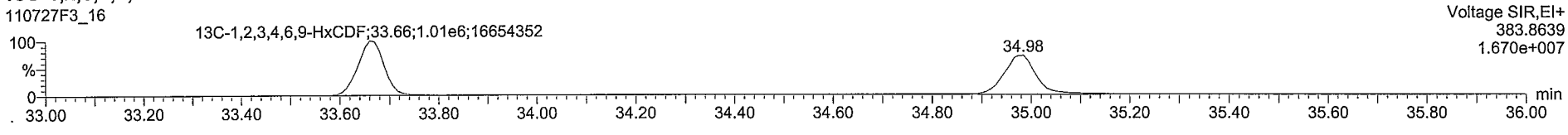
13C-1,2,3,7,8,9-HxCDD



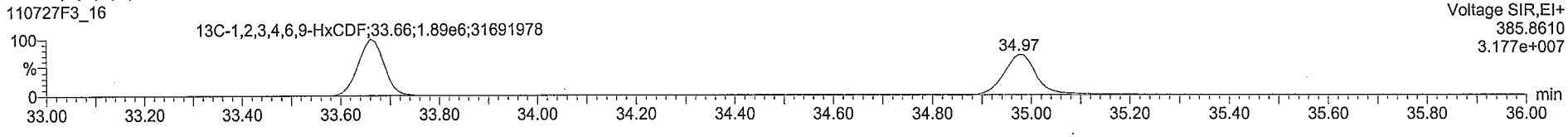
13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,4,6,9-HxCDF



13C-1,2,3,4,6,9-HxCDF



Dataset: C:\MassLynx\Default.pro\Results\110727F3\110727F3-17.qld

Last Altered: Thursday, July 28, 2011 11:45:48 Pacific Standard Time
Printed: Thursday, July 28, 2011 11:47:03 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-7-27-11.cdb 28 Jul 2011 08:40:50

Name: 110727F3_17, Date: 28-Jul-2011, Time: 03:31:17, ID: 27592_3913_014 MDL-SO-7 10, Description: MDL-SO-7

	# Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1 1,2,4,5,7,8-HCX	1.08e5	1.25	NO	0.210	10.000	37.25	36.208		0.403
2	2 13C-1,2,3,7,8,9-HxCDF	2.85e6	0.52	NO	0.874	10.000	34.97	209.76	105	0.147
3	3 13C-1,2,3,7,8,9-HxCDD	2.49e6	1.23	NO	0.799	10.000	34.57	200.38	100	0.145
4	4 13C-1,2,3,4,6,9-HxCDF	3.11e6	0.53	NO	1.00	10.000	33.66	200.00	100	0.129
5	5 PFK1					1.000				

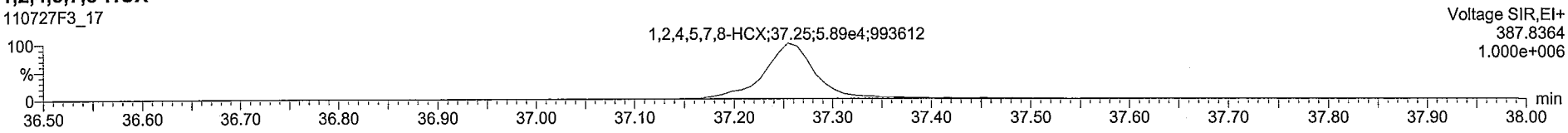
Fes 7/28/11

Dataset: Untitled

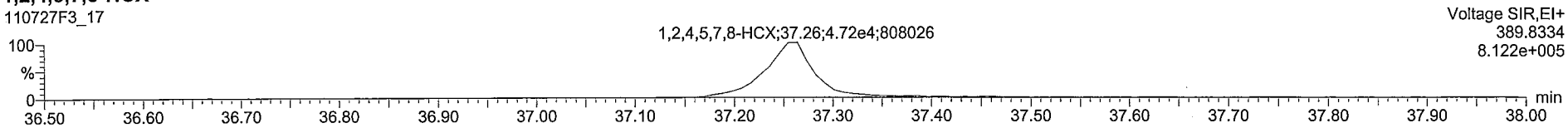
Last Altered: Thursday, July 28, 2011 09:26:41 Pacific Standard Time
Printed: Thursday, July 28, 2011 09:27:31 Pacific Standard Time

Name: 110727F3_17, Date: 28-Jul-2011, Time: 03:31:17, ID: 27592_3913_014 MDL-SO-7 10, Description: MDL-SO-7

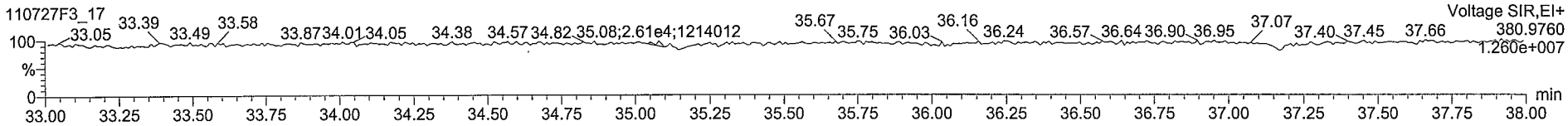
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1

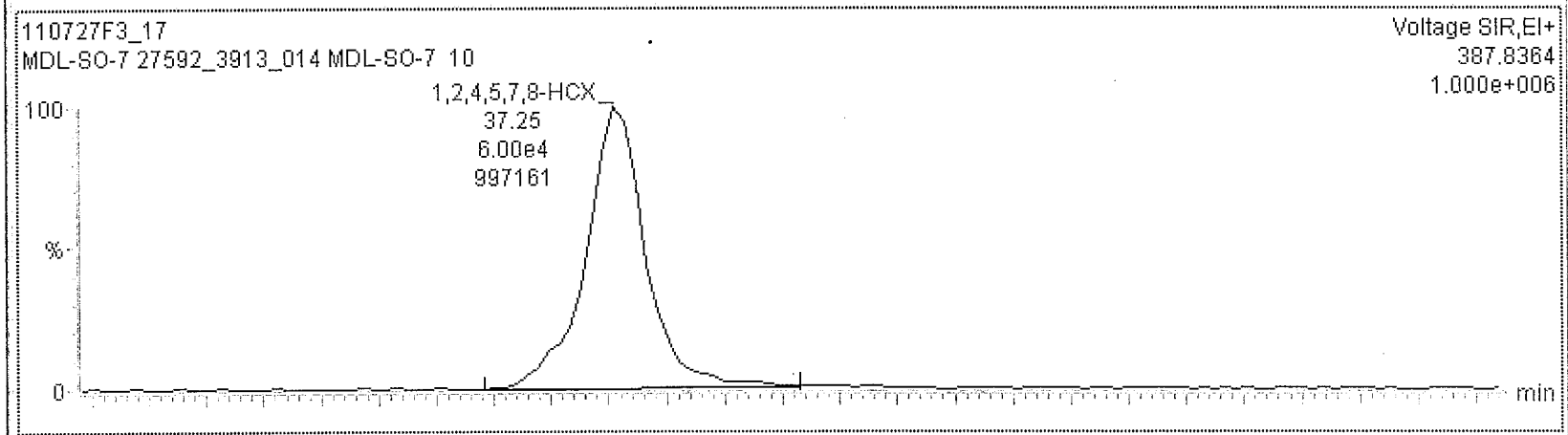


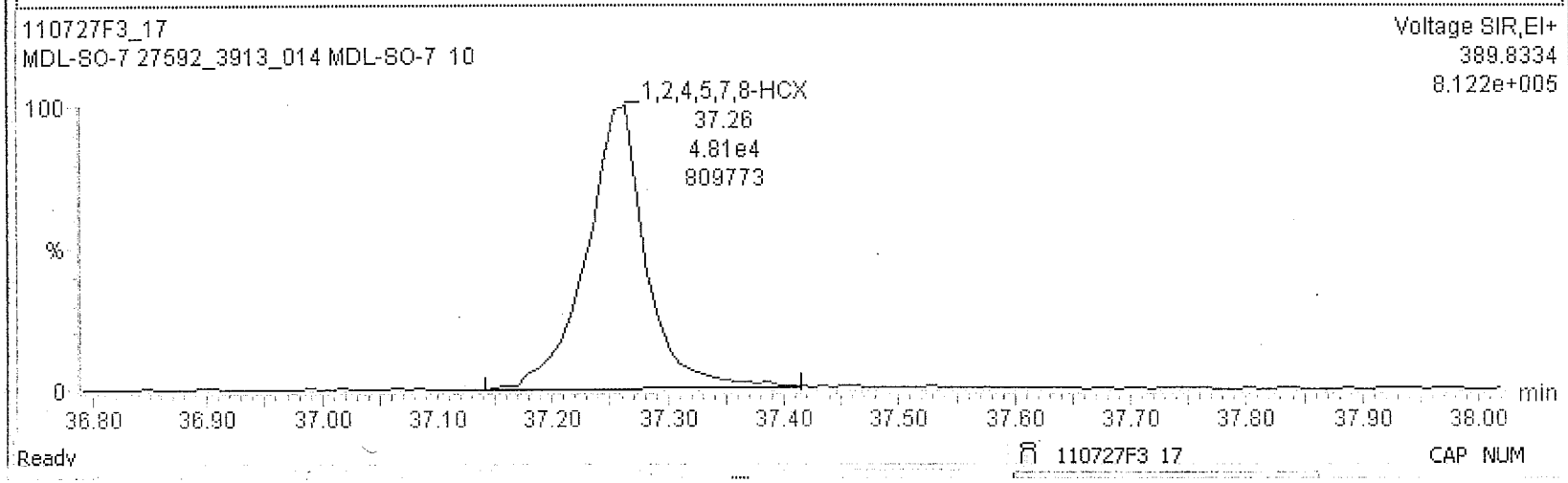
TargetLynx - untitled * - [Chromatogram]

File Edit View Display Processing Window Help

110727F3_17 27592_3913_014 MDL-SO-7 10 MDL-SO-7

	#	Name	Resp	RA	n/y	RRF	wt/vol	RT	Conc.	%Rec	DL
1	1	1,2,4,5,7,8-HCX	1.08e5	1.25	NO	0.21	10.000	37.25	36.2		0.403
2	2	13C-1,2,3,7,8,9-HxCDF	2.85e6	0.52	NO	0.87	10.000	34.97	210	105	0.147
3	3	13C-1,2,3,7,8,9-HxCDD	2.49e6	1.23	NO	0.80	10.000	34.57	200	100	0.145
4	4	13C-1,2,3,4,6,9-HxCDF	3.11e6	0.53	NO	1.00	10.000	33.66	200	100	0.129
5	5	PFK1					1.000				





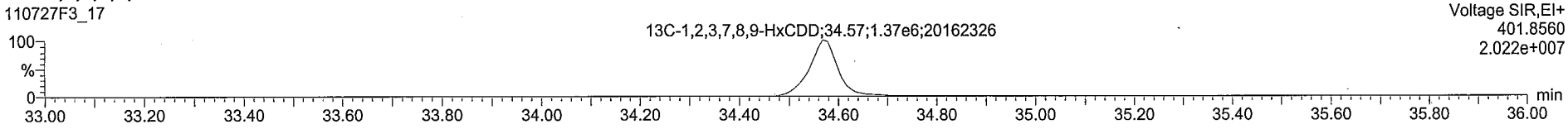
Ready 110727F3 17 CAP NUM

Dataset: Untitled

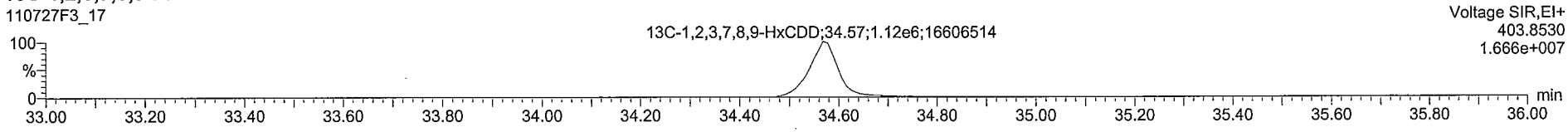
Last Altered: Thursday, July 28, 2011 09:26:41 Pacific Standard Time
Printed: Thursday, July 28, 2011 09:27:31 Pacific Standard Time

Name: 110727F3_17, Date: 28-Jul-2011, Time: 03:31:17, ID: 27592_3913_014 MDL-SO-7 10, Description: MDL-SO-7

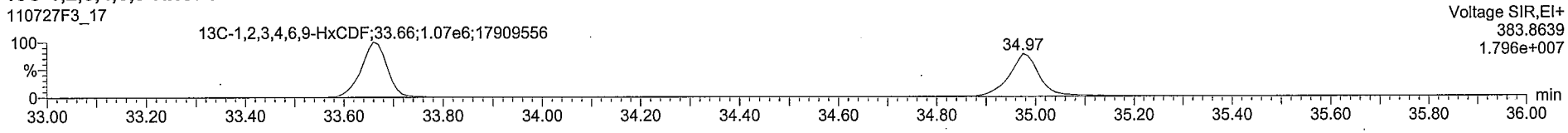
13C-1,2,3,7,8,9-HxCDD



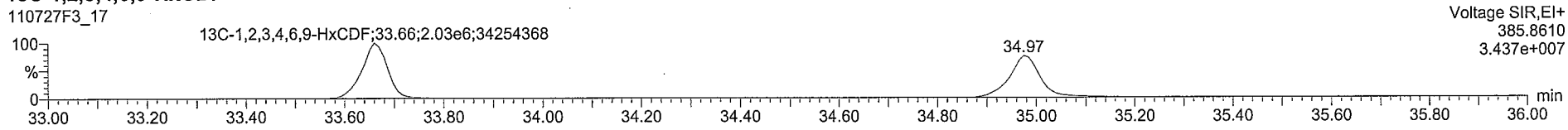
13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,4,6,9-HxCDF



13C-1,2,3,4,6,9-HxCDF

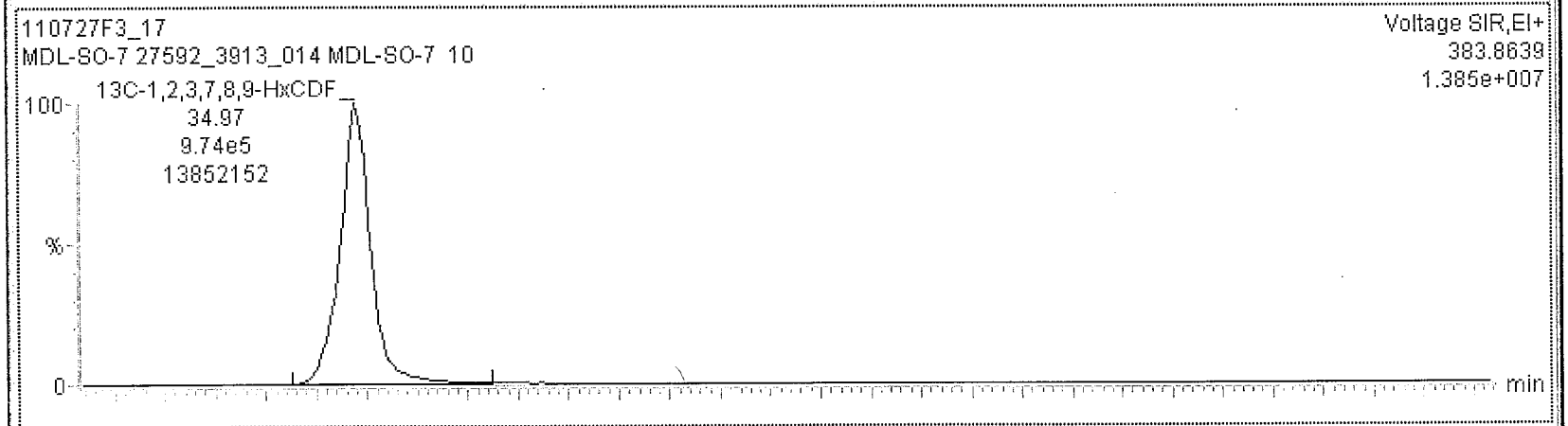


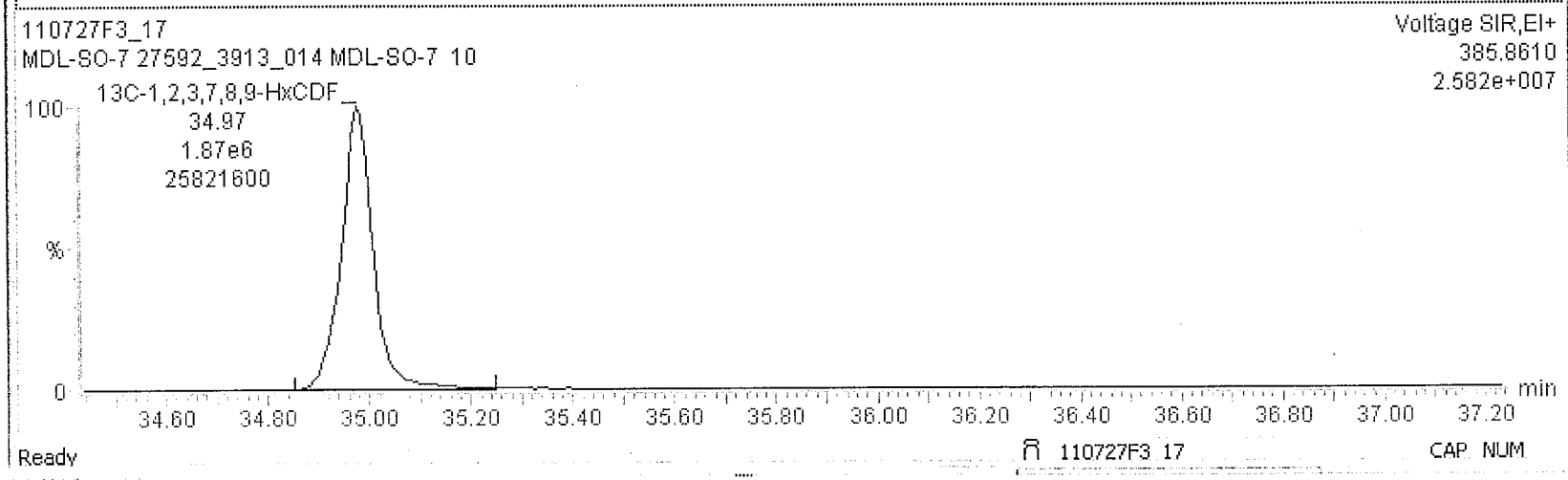
TargetLynx - untitled * - [Chromatogram]

File Edit View Display Processing Window Help

110727F3_17_27592_3913_014 MDL-SO-7 10 MDL-SO-7

	#	Name	Resp	RA	n/y	RRF	wt/vol	RT	Conc.	%Rec	DL
1	1	1,2,4,5,7,8-HCX	1.08e5	1.25	NO	0.21	10.000	37.25	36.2		0.403
2	2	13C-1,2,3,7,8,9-HxCDF	2.85e6	0.52	NO	0.87	10.000	34.97	210	105	0.147
3	3	13C-1,2,3,7,8,9-HxCDD	2.49e6	1.23	NO	0.80	10.000	34.57	200	100	0.145
4	4	13C-1,2,3,4,6,9-HxCDF	3.11e6	0.53	NO	1.00	10.000	33.66	200	100	0.129
5	5	PFK1					1.000				





Ready

110727F3_17

CAP NUM

Attachment F

MDL Study Liquid (Full Laboratory Deliverable)

MDL
Hexachloroxanthene

HCX

2-Aug-11

	Amt. Spiked	1	2	3	4	5	6	7	Ave	SD	MDL	RL
Hexachloroxanthene	500	287	307	300	289	286	315	294	296.86	11.01	34.59	50

Batch: 3922

Units: pg/L

PROCESS SHEET

Project No.-AR: 27592-251 of 257

Prep Due: 7/16/2011 **Project Due:** 7/14/2011

Hold Due: 5/10/2012 **TAT:** 21

Client: Vista Analytical Laboratory(AALCA01D)

Client Manager: Christina A. Vredevoe

Method: Hexachlorophene | Hexachlorophene
Hexachloroxanthene | Hexachloroxanthene

Split Type: 3922

Matrix: Aqueous

LabID	Recon	Client-ID	Description	Date Received	SLoc	Shelf
001	☐	MDL-AQ-1		7/25/2011	WR-2	
002	☐	MDL-AQ-2		7/25/2011	WR-2	
003	☐	MDL-AQ-3		7/25/2011	WR-2	
004	☐	MDL-AQ-4		7/25/2011	WR-2	
005	☐	MDL-AQ-5		7/25/2011	WR-2	
006	☐	MDL-AQ-6		7/25/2011	WR-2	
007	☐	MDL-AQ-7		7/25/2011	WR-2	

Instructions:
HCX only - no split

Report Options

Report Level:

TEQ Type:

EDD Type:

Report Group: Dioxins NoMDL No %Solid

Samples Reconciled By: N/A / /

Vial Box ID: _____

Date Requested 7/3/2011

Project: 27592

Extraction Set: 3922

Chemist: C. Vredeveld 7/31/11

Method(s): Hexachlorophene/Hexachloroxanthene | Hexachlorophene/Hexachloroxanthene

Prep time: 1018

C	VISTA Sample ID	G Eqv	Sample Amt. (L)	IS/NS CHEM/ WIT DATE	CRS CHEM/WIT DATE	AP CHEM/Date	SG ~~ABSG~~ CHEM/Date	AA CHEM/Date	Florisil CHEM/Date	RS CHEM/WIT DATE
☐	0_3922_MB001	N/A	(1.000)	CV AR 7/31/11	CV AR 7/31/11	N/A	CV 7/31/11	N/A	N/A	CV AR 7/31/11
☐	27592_3922_001	↓	↓	↓	↓	↓	↓	↓	↓	↓
☐	27592_3922_002									
☐	27592_3922_003									
☐	27592_3922_004									
☐	27592_3922_005									
☐	27592_3922_006									
☐	27592_3922_007									

IS Name	NS Name	CRS Name	RS Name
	NDL		
PCDD/F	PCDD/F	PCDD/F	PCDD/F
PCB	PCB 110713A 25µL	PCB	PCB
HCx 110706A 10µL	HCx ~~H0701A 10µL~~ CV 7/31/11	HCx 110503B 10µL	HCx 110503C 10µL

Cycle Time
Start: N/A
Stop: ↓

APP.: (SEFUN) SOX SDS
SOLV: DCM
Other: N/A
Final Volume(s): 20µL CV

Check Out:
Chemist: N/A / /
Check-In:
Chemist: / /
Balance ID:

Comments:

CALIBRATION STANDARDS REVIEW CHECKLIST

Vista Analytical Laboratory

Beg. Calibration ID: ST110802F2-1

End Calibration ID: N/A

	Beg.	End
Ion abundance within QC limits?	✓	NA
Concentration within range?	✓	
First and last eluters present?	NA	
Retention Times within criteria?	NA	
Verification Std. named correctly? (ST-Year-Month-Day-VG ID)	✓	
Forms signed and dated?	✓	
Correct ICAL referenced?	✓	
Run Log:		
-Data file matches Conc Cal ID?	✓	
-Correct instrument listed?	✓	
-Samples within 12-hour clock?	(y)	n

	Beg.	End
Mass resolution > 10,000? ▪ Method 1614 > 5,000; CARB 429 > 8,000	✓	✓
TCDD/TCDF valleys < 25%?	NA	N/A
Peaks integrated correctly?	✓	
Manual integrations included?		
8280 CS1 Ending Standard		
-Ratios within limits		
-S/N > 2.5:1		
-CS1 within 12-hour clock		✓

Comments:

Reviewed by: DMS 8/3/11
Initials & Date

* *Ending standard criteria applicable to 8290 only.*

Vista Analytical Laboratory VG-9

Dataset: Untitled

Last Altered: Wednesday, August 03, 2011 12:15:31 PM Pacific Standard Time
Printed: Wednesday, August 03, 2011 12:15:52 PM Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-8-01-11.cdb 02 Aug 2011 09:02:25

Compound name: 1,2,4,5,7,8-HCX

	Name	ID	Acq.Date	Acq.Time
1	110802F2_1	ST110802F2-1 HCX CS3 110706E	02-Aug-11	15:07:04
2	110802F2_2	SOLVENT BLANK	02-Aug-11	15:49:08
3	110802F2_3	0_3922_MB001	02-Aug-11	16:32:46
4	110802F2_4	0_3923_MB001	02-Aug-11	17:16:23
5	110802F2_5	27592_3922_001 MDL-AQ-1 1	02-Aug-11	18:00:02
6	110802F2_6	27592_3922_002 MDL-AQ-2 1	02-Aug-11	18:43:40
7	110802F2_7	27592_3922_003 MDL-AQ-3 1	02-Aug-11	19:27:13
8	110802F2_8	27592_3922_004 MDL-AQ-4 1	02-Aug-11	20:10:51
9	110802F2_9	27592_3922_005 MDL-AQ-5 1	02-Aug-11	20:54:18
10	110802F2_10	27592_3922_006 MDL-AQ-6 1	02-Aug-11	21:37:56
11	110802F2_11	27592_3922_007 MDL-AQ-7 1	02-Aug-11	22:21:34
12	110802F2_12	27592_3923_043 IPR-Aqueous-1 1	02-Aug-11	23:05:08
13	110802F2_13	27592_3923_044 IPR-Aqueous-2 1	02-Aug-11	23:48:41
14	110802F2_14	27592_3923_045 IPR-Aqueous-3 1	03-Aug-11	00:32:14
15	110802F2_15	27592_3923_046 IPR-Aqueous-4 1	03-Aug-11	01:15:51
16	110802F2_16	SOLVENT BLANK	03-Aug-11	01:59:29

Dataset: C:\MassLynx\Default.pro\Results\110802F2\110802F2_3.qld

Last Altered: Wednesday, August 03, 2011 12:11:12 PM Pacific Standard Time
Printed: Wednesday, August 03, 2011 12:12:15 PM Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-8-01-11.cdb 02 Aug 2011 09:02:25

Name: 110802F2_3, Date: 02-Aug-2011, Time: 16:32:46, ID: 0_3922_MB001, Description: 0_3922_MB001

	#	Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1	1,2,4,5,7,8-HCX				0.220	1.000				12.9
2	2	13C-1,2,3,7,8,9-HxCDF	1.17e6	0.53	NO	0.869	1.000	34.97	1815.5	90.8	2.14
3	3	13C-1,2,3,7,8,9-HxCDD	1.03e6	1.23	NO	0.717	1.000	34.56	1925.9	96.3	2.74
4	4	13C-1,2,3,4,6,9-HxCDF	1.49e6	0.53	NO	1.00	1.000	33.66	2000.0	100	1.86

FEB 8/3/11

Vista Analytical Laboratory VG-9

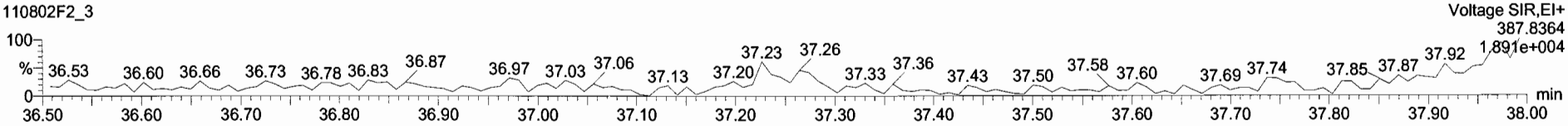
Dataset: Untitled

Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

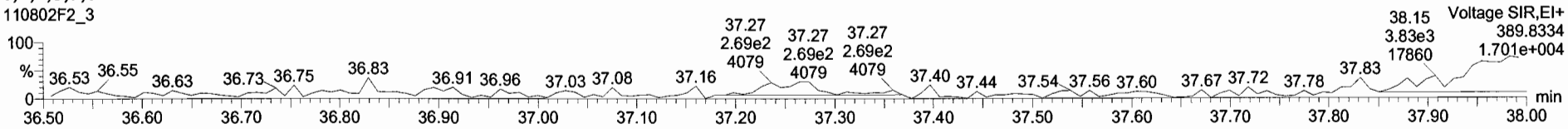
Method: C:\MassLynx\Default.pro\Methdb\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\Default.pro\Curvedb\db-5_hcxvg9-8-01-11.cdb 02 Aug 2011 09:02:25

Name: 110802F2_3, Date: 02-Aug-2011, Time: 16:32:46, ID: 0_3922_MB001, Description: 0_3922_MB001

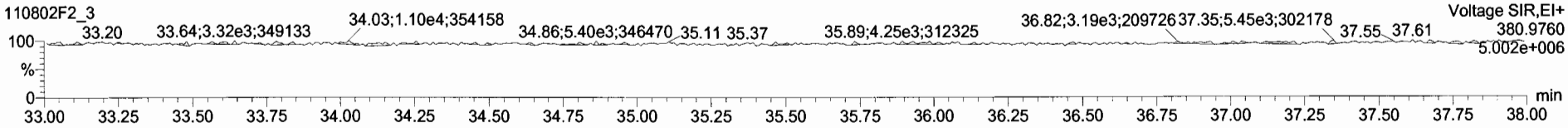
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1

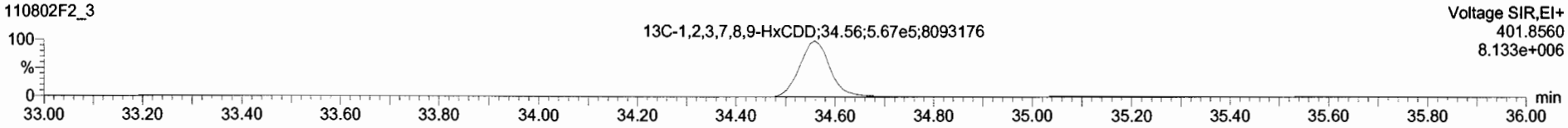


Dataset: Untitled

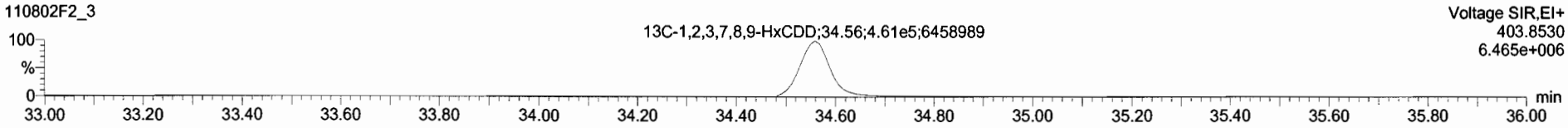
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_3, Date: 02-Aug-2011, Time: 16:32:46, ID: 0_3922_MB001, Description: 0_3922_MB001

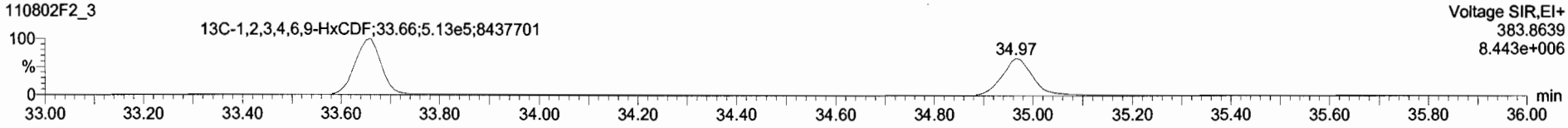
13C-1,2,3,7,8,9-HxCDD



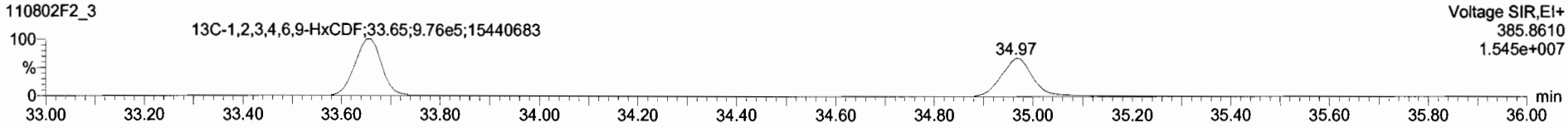
13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,4,6,9-HxCDF



13C-1,2,3,4,6,9-HxCDF



Dataset: C:\MassLynx\Default.pro\Results\110802F2\110802F2_5.qld

Last Altered: Wednesday, August 03, 2011 12:23:10 Pacific Standard Time
Printed: Wednesday, August 03, 2011 12:23:57 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-8-01-11.cdb 02 Aug 2011 09:02:25

Name: 110802F2_5, Date: 02-Aug-2011, Time: 18:00:02, ID: 27592_3922_001 MDL-AQ-1 1, Description: MDL-AQ-1

	# Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1 1,2,4,5,7,8-HCX	3.61e4	1.26	NO	0.220	1.000	37.24	287.33		35.4
2	2 13C-1,2,3,7,8,9-HxCDF	1.14e6	0.52	NO	0.869	1.000	34.96	1784.2	89.2	2.78
3	3 13C-1,2,3,7,8,9-HxCDD	1.10e6	1.26	NO	0.717	1.000	34.55	2080.3	104	2.89
4	4 13C-1,2,3,4,6,9-HxCDF	1.47e6	0.53	NO	1.00	1.000	33.65	2000.0	100	2.42

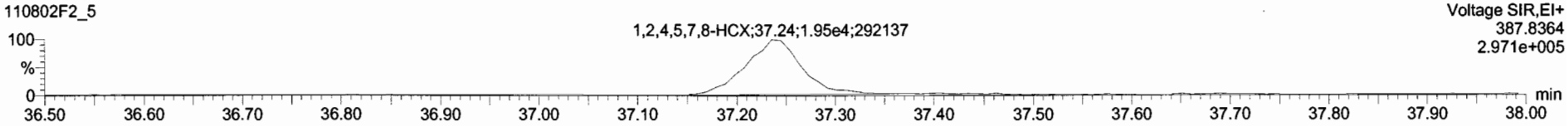
FEB 8/3/11

Dataset: Untitled

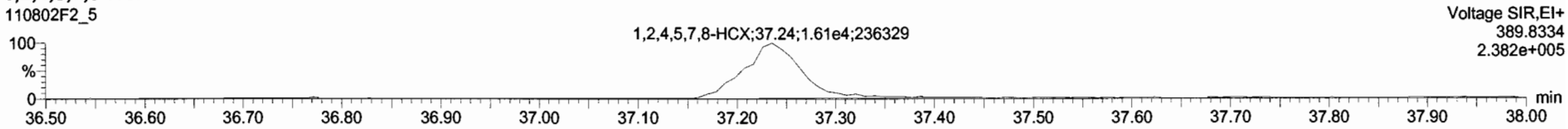
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_5, Date: 02-Aug-2011, Time: 18:00:02, ID: 27592_3922_001 MDL-AQ-1 1, Description: MDL-AQ-1

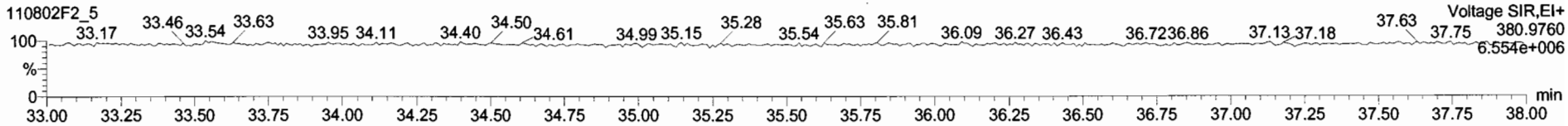
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1



TargetLynx - Untitled - [Chromatogram]

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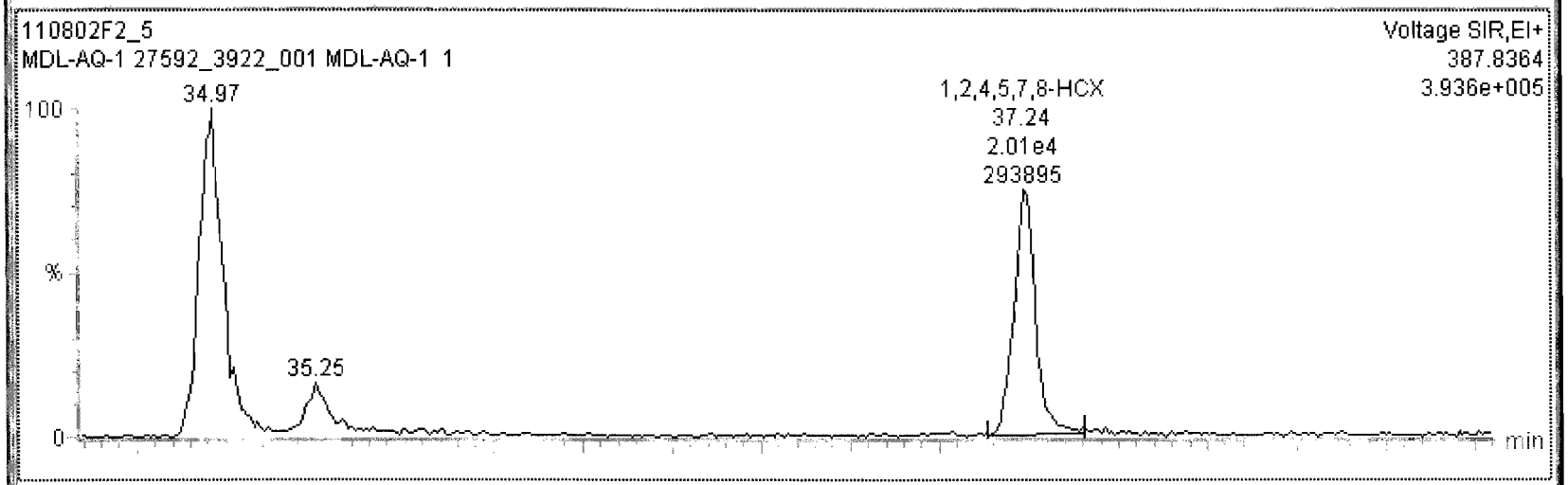
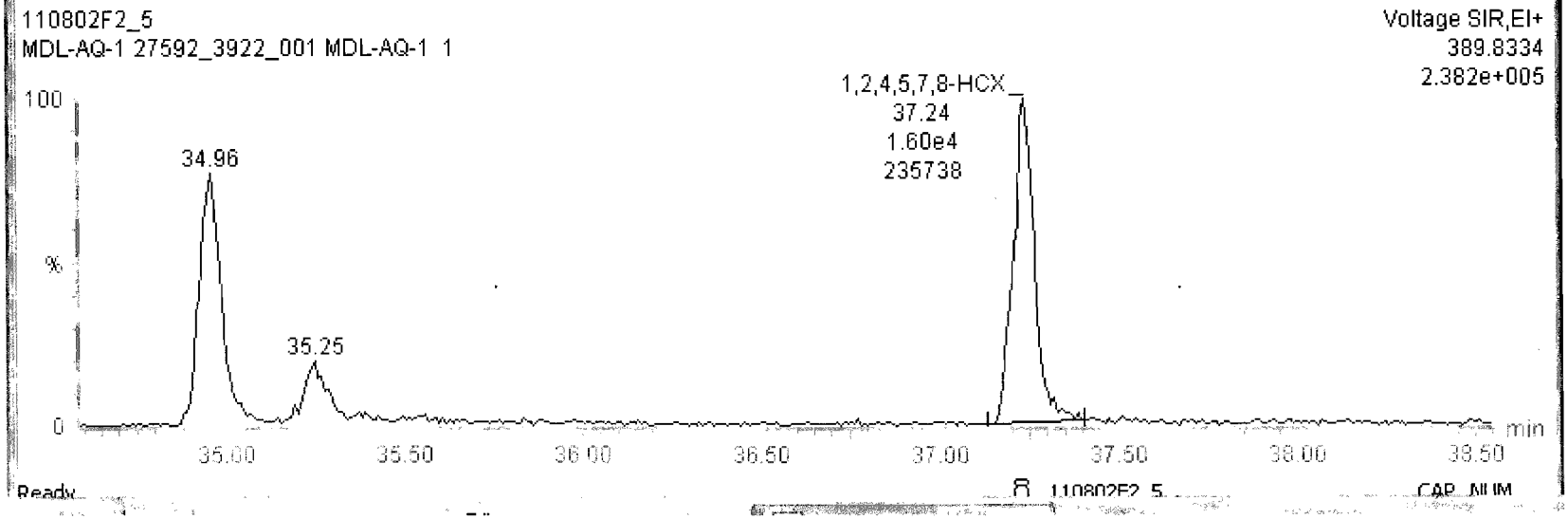
110802F2_5 - 27592_3922_001 MDL-AQ-1 1 - MDL-AQ-1

	#	Name	Resp	RA	n/y	RRF	wt/vol	RT	Conc.	%Rec	DL
1	1	1,2,4,5,7,8-HCX	3.61e4	1.26	NO	0.22	1.000	37.24	287		35.4
2	2	13C-1,2,3,7,8,9-HxCDF	1.14e6	0.52	NO	0.87	1.000	34.96	1780	89.2	2.78
3	3	13C-1,2,3,7,8,9-HxCDD	1.10e6	1.26	NO	0.72	1.000	34.55	2080	104	2.89
4	4	13C-1,2,3,4,6,9-HxCDF	1.47e6	0.53	NO	1.00	1.000	33.65	2000	100	2.42
5	5	PFK1					1.000				





Ready

110802F2_5

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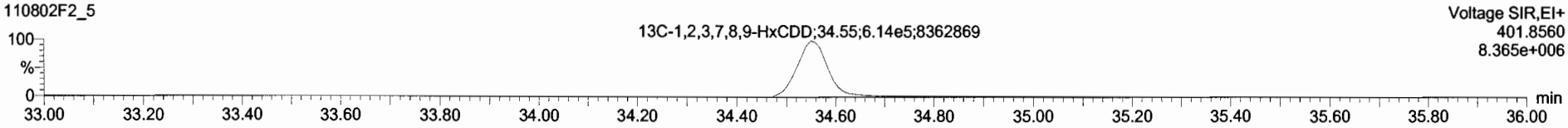
Dataset: Untitled

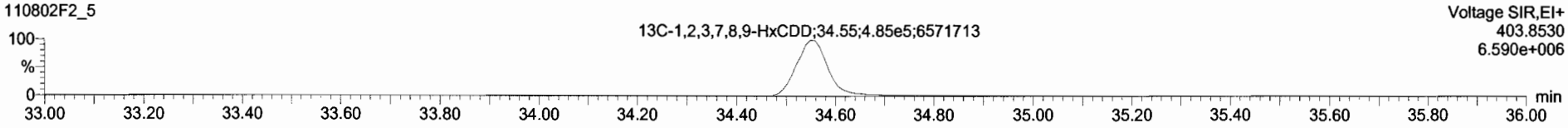
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_5, Date: 02-Aug-2011, Time: 18:00:02, ID: 27592_3922_001 MDL-AQ-1 1, Description: MDL-AQ-1

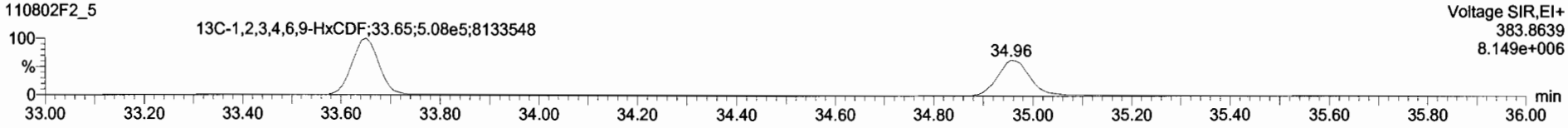
13C-1,2,3,7,8,9-HxCDD



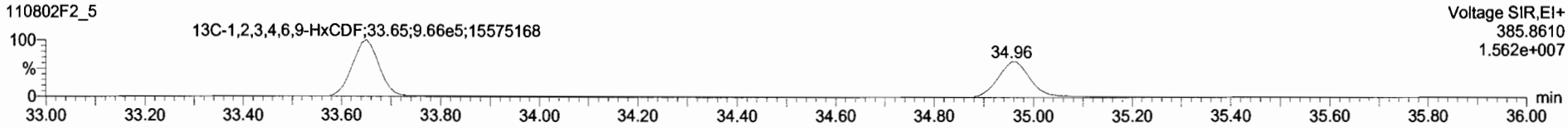
13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,4,6,9-HxCDF



13C-1,2,3,4,6,9-HxCDF



Dataset: C:\MassLynx\Default.pro\Results\110802F2\110802F2_6.qld

Last Altered: Wednesday, August 03, 2011 12:25:52 Pacific Standard Time
Printed: Wednesday, August 03, 2011 12:26:45 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-8-01-11.cdb 02 Aug 2011 09:02:25

Name: 110802F2_6, Date: 02-Aug-2011, Time: 18:43:40, ID: 27592_3922_002 MDL-AQ-2 1, Description: MDL-AQ-2

	#	Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1	1,2,4,5,7,8-HCX	3.77e4	1.24	NO	0.220	1.000	37.25	306.71		7.95
2	2	13C-1,2,3,7,8,9-HxCDF	1.12e6	0.52	NO	0.869	1.000	34.96	1935.4	96.8	2.59
3	3	13C-1,2,3,7,8,9-HxCDD	1.04e6	1.25	NO	0.717	1.000	34.56	2172.3	109	4.15
4	4	13C-1,2,3,4,6,9-HxCDF	1.33e6	0.53	NO	1.00	1.000	33.65	2000.0	100	2.25

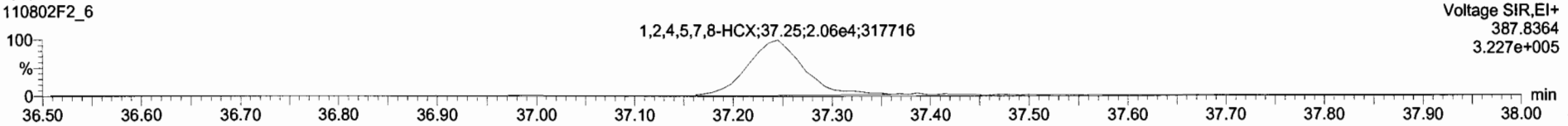
FEB 8 | 3/11

Dataset: Untitled

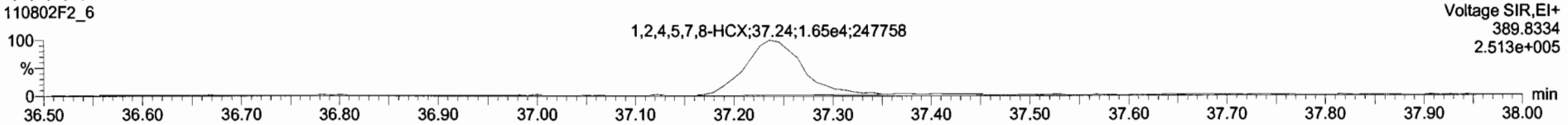
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_6, Date: 02-Aug-2011, Time: 18:43:40, ID: 27592_3922_002 MDL-AQ-2 1, Description: MDL-AQ-2

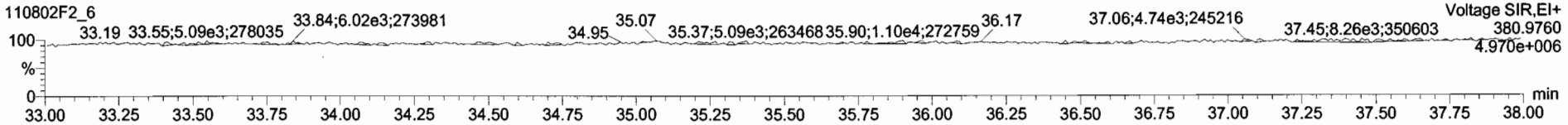
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1



TargetLynx - untitled - [Chromatogram]

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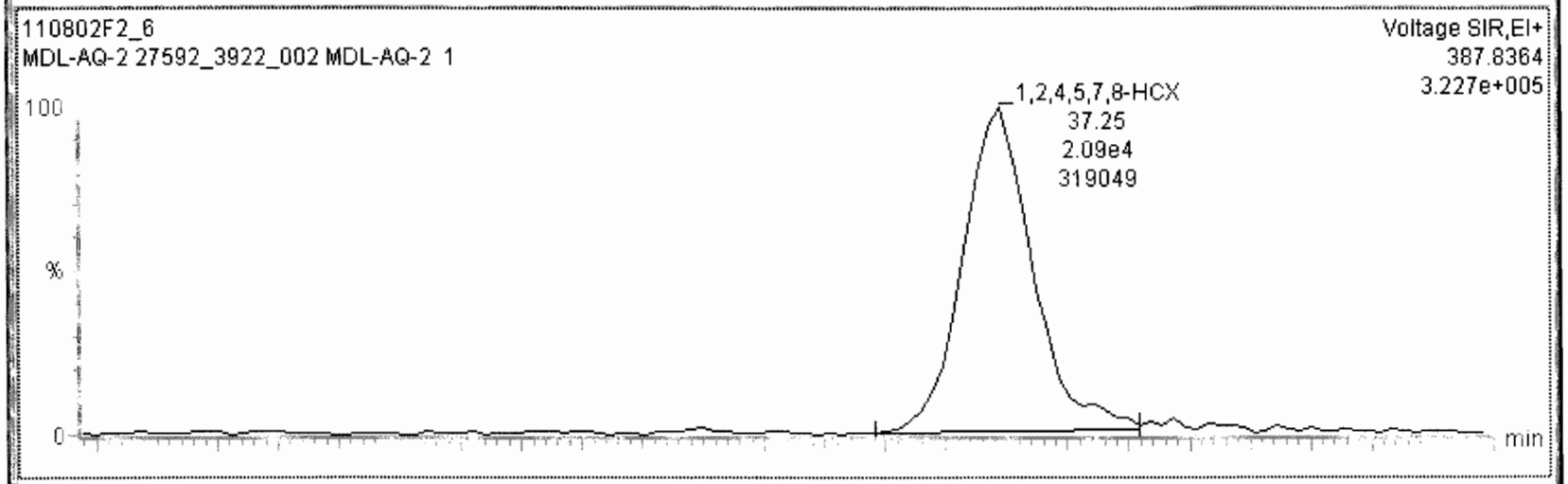
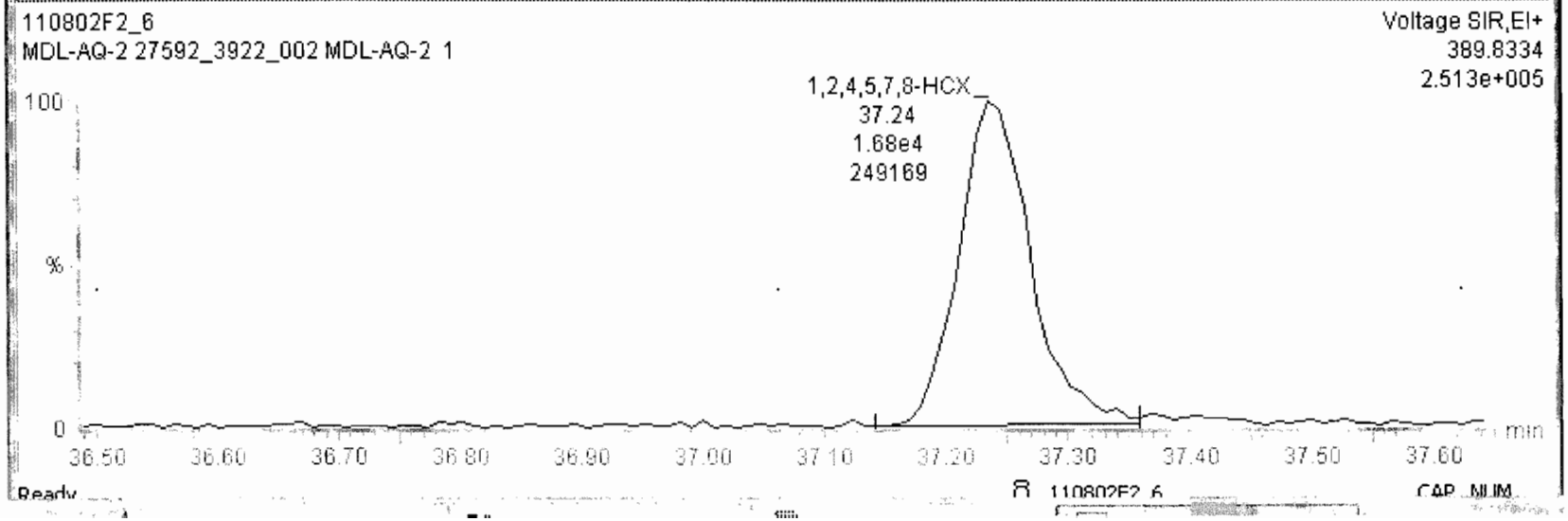
110802F2_6 - 27592_3922_002 MDL-AQ-2 1 - MDL-AQ-2

	#	Name	Resp	RA	n/y	RRF	wt/vol	RT	Conc.	%Rec	DL
1	1	1,2,4,5,7,8-HCX	3.77e4	1.24	NO	0.22	1.000	37.25	307		7.95
2	2	13C-1,2,3,7,8,9-HxCDF	1.12e6	0.52	NO	0.87	1.000	34.96	1940	96.8	2.59
3	3	13C-1,2,3,7,8,9-HxCDD	1.04e6	1.25	NO	0.72	1.000	34.56	2170	109	4.15
4	4	13C-1,2,3,4,6,9-HxCDF	1.33e6	0.53	NO	1.00	1.000	33.65	2000	100	2.25
5	5	PFK1					1.000				



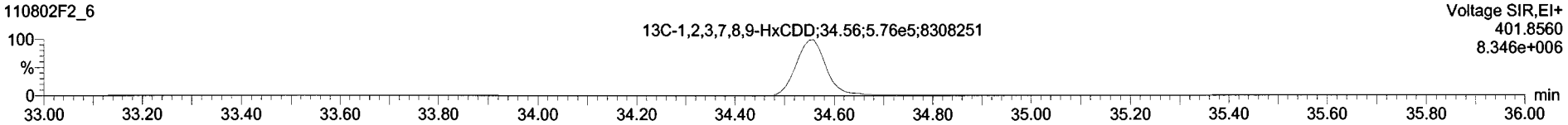


Dataset: Untitled

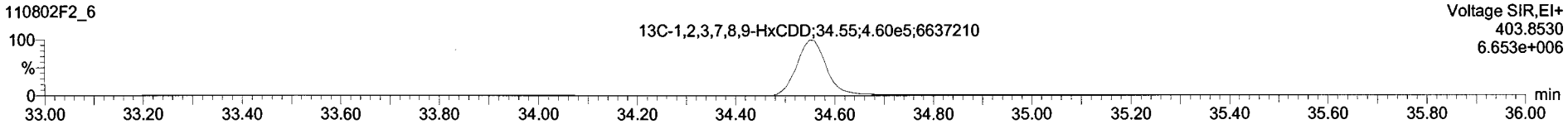
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_6, Date: 02-Aug-2011, Time: 18:43:40, ID: 27592_3922_002 MDL-AQ-2 1, Description: MDL-AQ-2

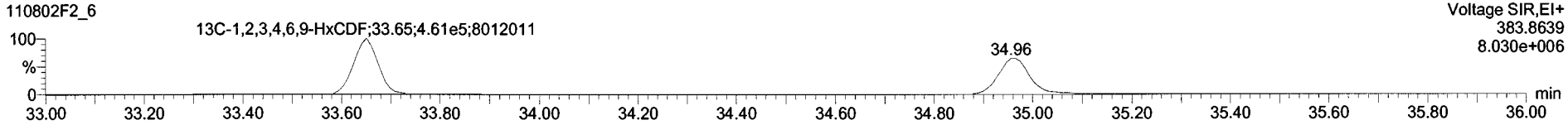
13C-1,2,3,7,8,9-HxCDD



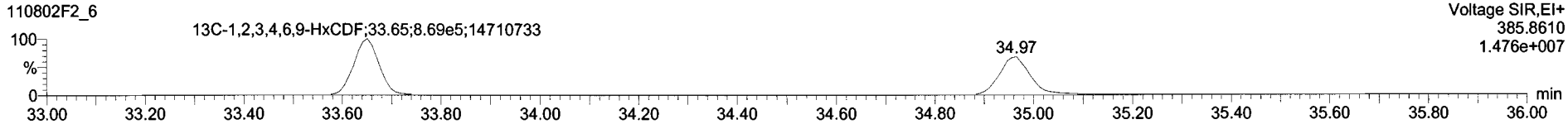
13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,4,6,9-HxCDF



13C-1,2,3,4,6,9-HxCDF



Dataset: C:\MassLynx\Default.pro\Results\110802F2\110802F2_7.qld

Last Altered: Wednesday, August 03, 2011 12:31:37 Pacific Standard Time
Printed: Wednesday, August 03, 2011 12:35:19 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-8-01-11.cdb 02 Aug 2011 09:02:25

Name: 110802F2_7, Date: 02-Aug-2011, Time: 19:27:13, ID: 27592_3922_003 MDL-AQ-3 1, Description: MDL-AQ-3

	# Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1 1,2,4,5,7,8-HCX	3.26e4	1.26	NO	0.220	1.000	37.24	300.05		9.06
2	2 13C-1,2,3,7,8,9-HxCDF	9.87e5	0.51	NO	0.869	1.000	34.97	1690.3	84.5	2.29
3	3 13C-1,2,3,7,8,9-HxCDD	1.02e6	1.26	NO	0.717	1.000	34.55	2125.0	106	2.90
4	4 13C-1,2,3,4,6,9-HxCDF	1.34e6	0.51	NO	1.00	1.000	33.65	2000.0	100	1.99

FEB 8/3/11

FEB 8/3/11

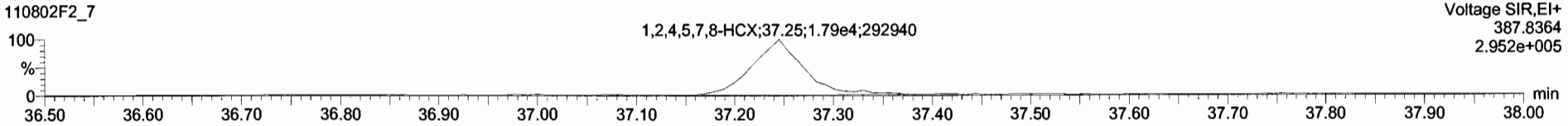
Vista Analytical Laboratory VG-9

Dataset: Untitled

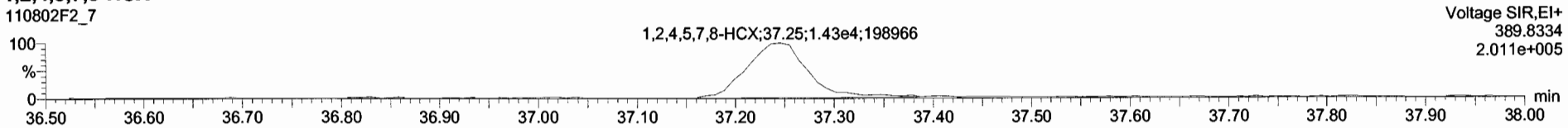
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_7, Date: 02-Aug-2011, Time: 19:27:13, ID: 27592_3922_003 MDL-AQ-3 1, Description: MDL-AQ-3

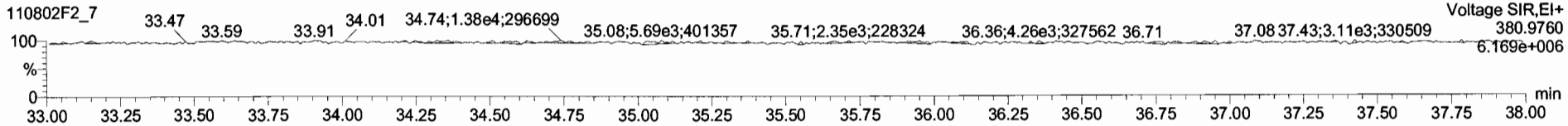
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1



TargetLynx - 110802F2_7 - [Chromatogram]

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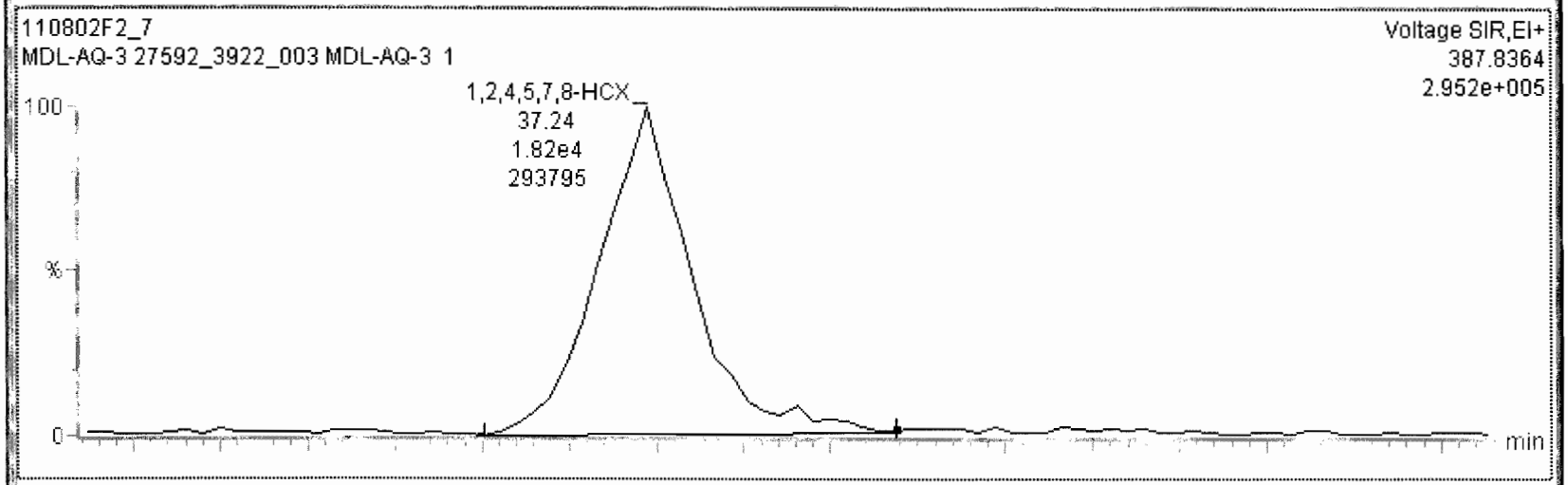
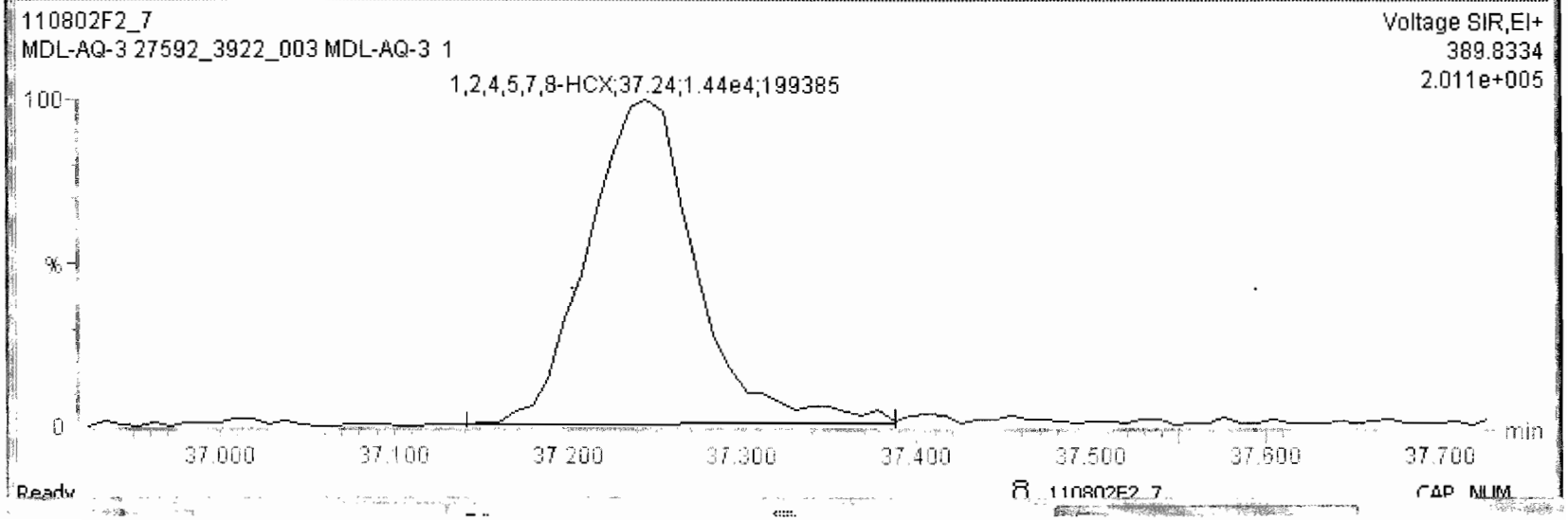
110802F2_7 - 27592_3922_003 MDL-AQ-3 1 - MDL-AQ-3

	#	Name	Resp	RA	n/y	RRF	wt/vol	RT	Conc.	%Rec	DL
1	1	1,2,4,5,7,8-HCX	3.26e4	1.26	NO	0.22	1.000	37.24	300		9.06
2	2	13C-1,2,3,7,8,9-HxCDF	9.87e5	0.51	NO	0.87	1.000	34.97	1690	84.5	2.29
3	3	13C-1,2,3,7,8,9-HxCDD	1.02e6	1.26	NO	0.72	1.000	34.55	2120	106	2.90
4	4	13C-1,2,3,4,6,9-HxCDF	1.34e6	0.51	NO	1.00	1.000	33.65	2000	100	1.99
5	5	PFK1					1.000				





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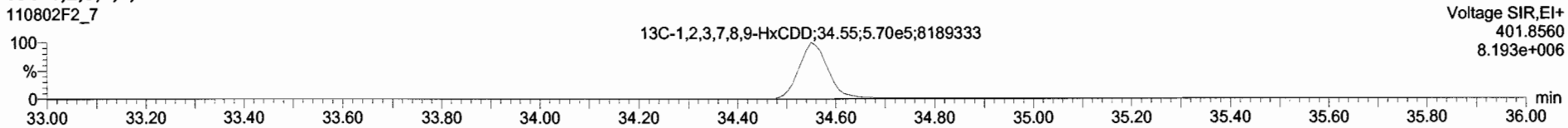
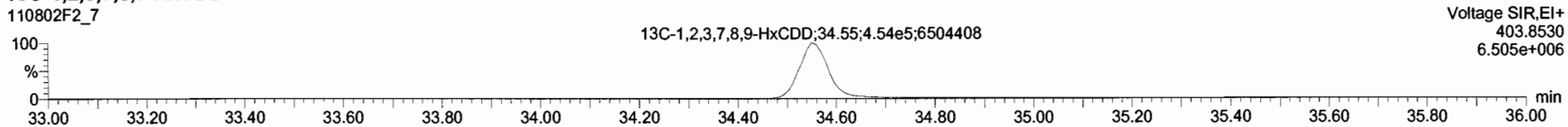
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_7, Date: 02-Aug-2011, Time: 19:27:13, ID: 27592_3922_003 MDL-AQ-3 1, Description: MDL-AQ-3

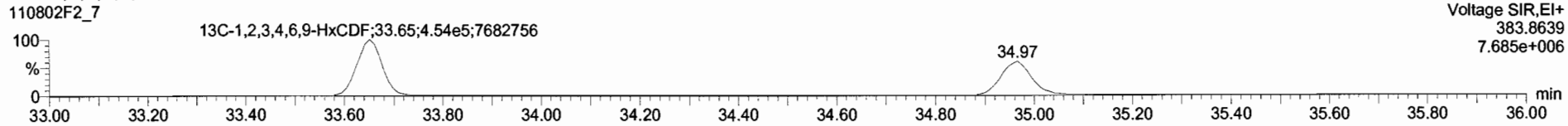
13C-1,2,3,7,8,9-HxCDD



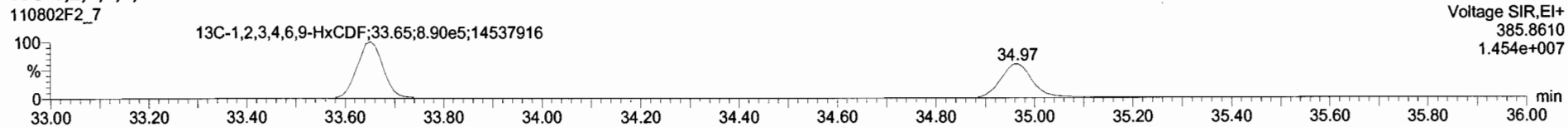
13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,4,6,9-HxCDF



13C-1,2,3,4,6,9-HxCDF



Dataset: C:\MassLynx\Default.pro\Results\110802F2\110802F2_8.qld

Last Altered: Wednesday, August 03, 2011 12:39:43 Pacific Standard Time
Printed: Wednesday, August 03, 2011 12:40:31 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-8-01-11.cdb 02 Aug 2011 09:02:25

Name: 110802F2_8, Date: 02-Aug-2011, Time: 20:10:51, ID: 27592_3922_004 MDL-AQ-4 1, Description: MDL-AQ-4

	# Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1 1,2,4,5,7,8-HCX	3.56e4	1.25	NO	0.220	1.000	37.25	289.15		8.81
2	2 13C-1,2,3,7,8,9-HxCDF	1.12e6	0.51	NO	0.869	1.000	34.97	1844.2	92.2	3.16
3	3 13C-1,2,3,7,8,9-HxCDD	1.03e6	1.25	NO	0.717	1.000	34.56	2059.0	103	2.39
4	4 13C-1,2,3,4,6,9-HxCDF	1.40e6	0.50	NO	1.00	1.000	33.65	2000.0	100	2.74

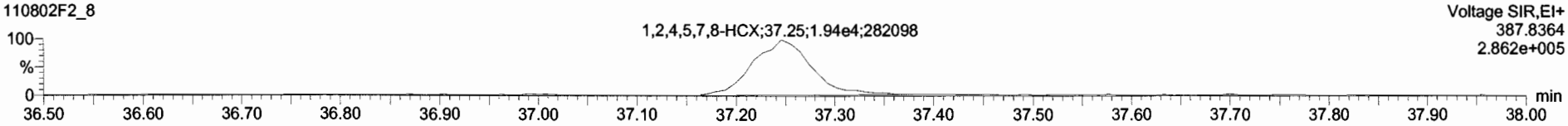
FEB 8/3/11

Dataset: Untitled

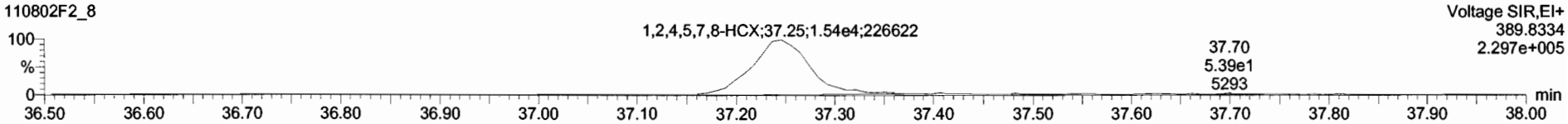
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_8, Date: 02-Aug-2011, Time: 20:10:51, ID: 27592_3922_004 MDL-AQ-4 1, Description: MDL-AQ-4

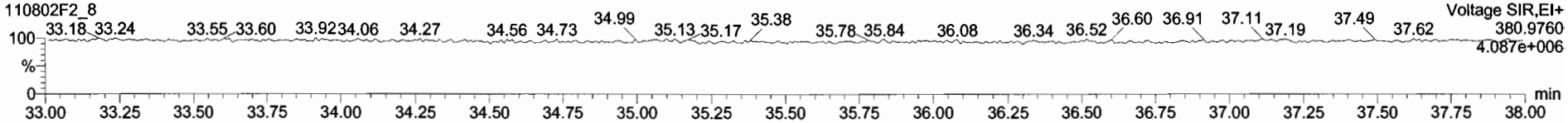
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1

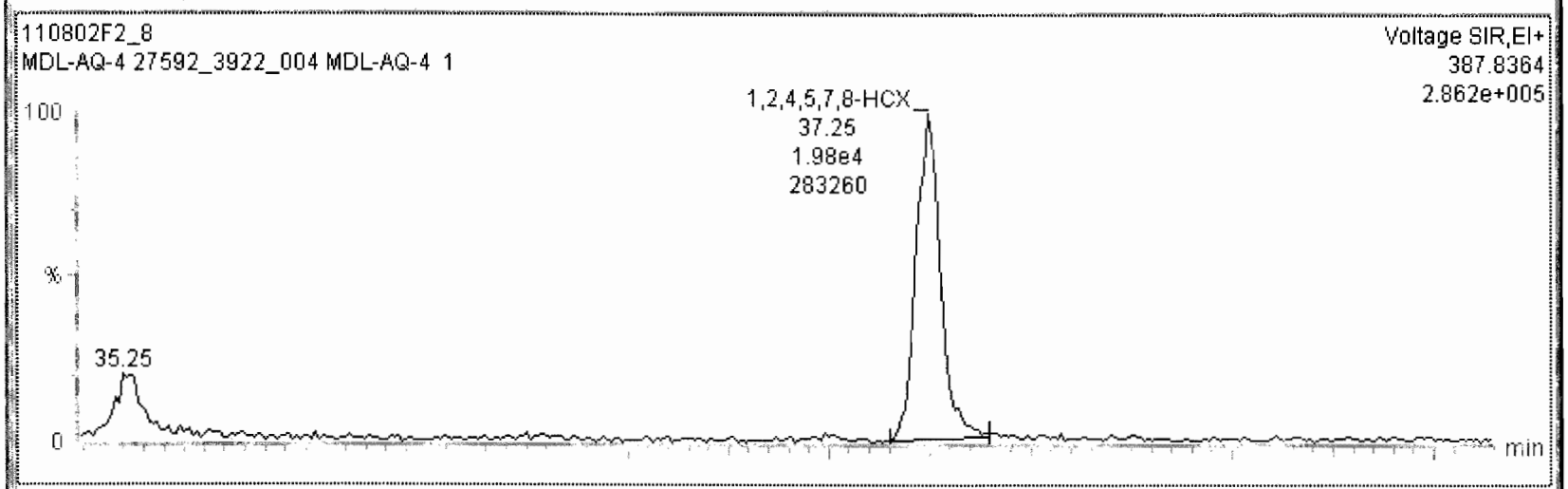


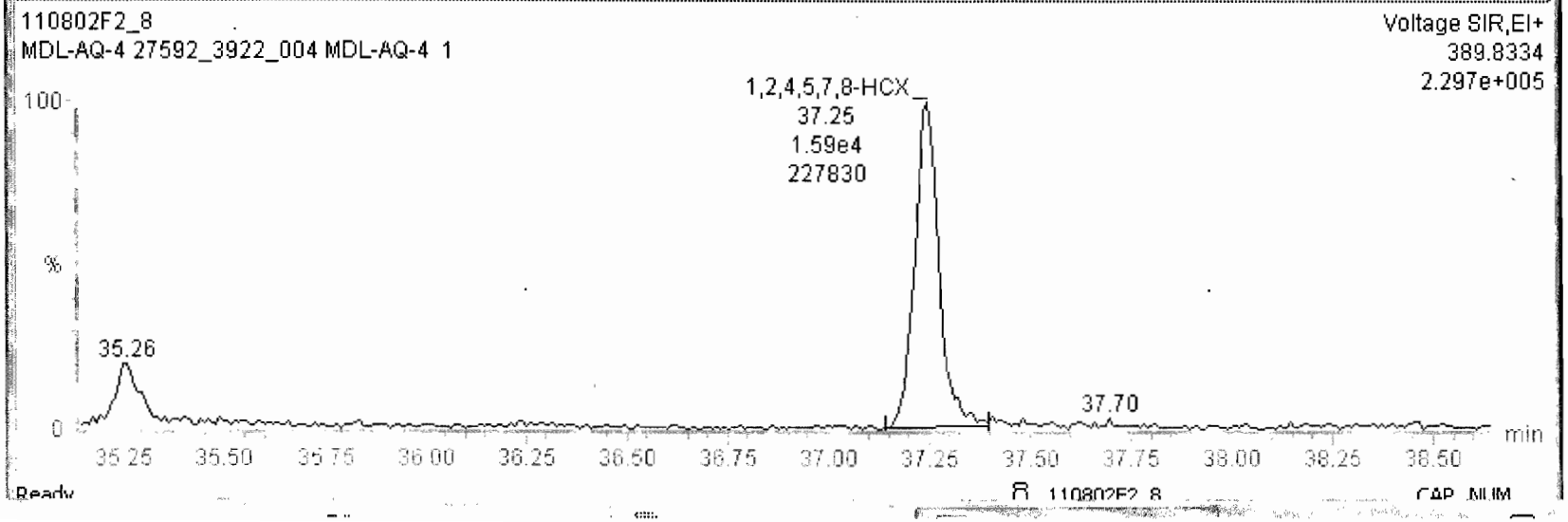
Target1.pnx - Untitled - [Chromatogram]

File Edit View Display Processing Window Help

110802F2_8 - 27592_3922_004 MDL-AQ-4 1 - MDL-AQ-4

	#	Name	Resp	RA	n/y	RRF	wt/vol	RT	Conc.	%Rec	DL
1	1	1,2,4,5,7,8-HCX	3.56e4	1.25	NO	0.22	1.000	37.25	289		8.81
2	2	13C-1,2,3,7,8,9-HxCDF	1.12e6	0.51	NO	0.87	1.000	34.97	1840	92.2	3.16
3	3	13C-1,2,3,7,8,9-HxCDD	1.03e6	1.25	NO	0.72	1.000	34.56	2060	103	2.39
4	4	13C-1,2,3,4,6,9-HxCDF	1.40e6	0.50	NO	1.00	1.000	33.65	2000	100	2.74
5	5	PFK1					1.000				



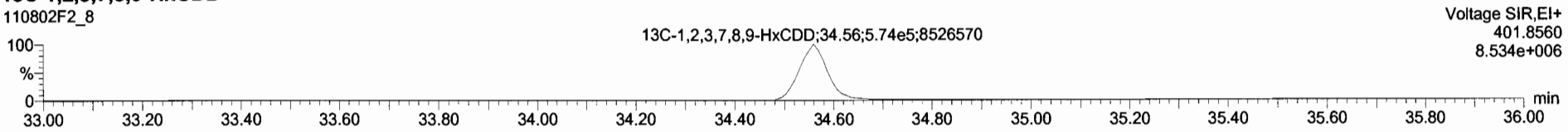


Dataset: Untitled

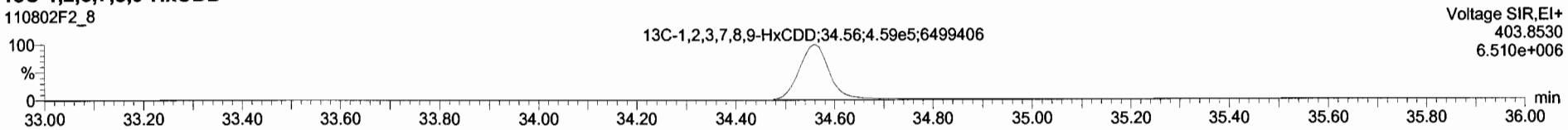
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_8, Date: 02-Aug-2011, Time: 20:10:51, ID: 27592_3922_004 MDL-AQ-4 1, Description: MDL-AQ-4

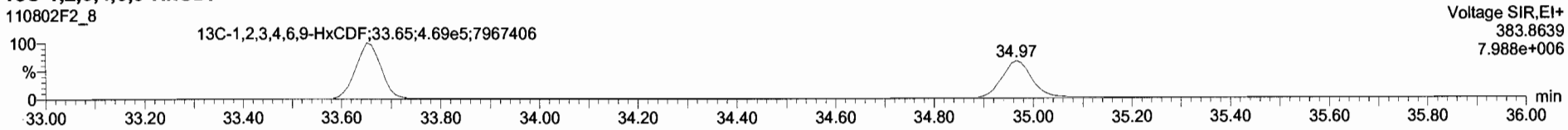
13C-1,2,3,7,8,9-HxCDD



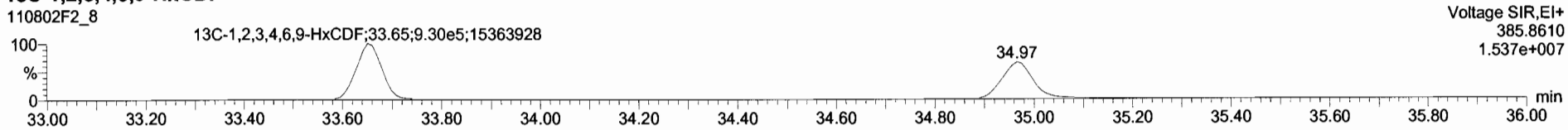
13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,4,6,9-HxCDF



13C-1,2,3,4,6,9-HxCDF



Dataset: C:\MassLynx\Default.pro\Results\110802F2\110802F2_9.qld

Last Altered: Wednesday, August 03, 2011 12:45:32 Pacific Standard Time
Printed: Wednesday, August 03, 2011 12:46:15 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-8-01-11.cdb 02 Aug 2011 09:02:25

Name: 110802F2_9, Date: 02-Aug-2011, Time: 20:54:18, ID: 27592_3922_005 MDL-AQ-5 1, Description: MDL-AQ-5

	# Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1 1,2,4,5,7,8-HCX	3.23e4	1.23	NO	0.220	1.000	37.26	286.04		9.96
2	2 13C-1,2,3,7,8,9-HxCDF	1.03e6	0.52	NO	0.869	1.000	34.97	1896.9	94.8	3.03
3	3 13C-1,2,3,7,8,9-HxCDD	9.15e5	1.24	NO	0.717	1.000	34.55	2050.3	103	3.00
4	4 13C-1,2,3,4,6,9-HxCDF	1.25e6	0.53	NO	1.00	1.000	33.65	2000.0	100	2.63

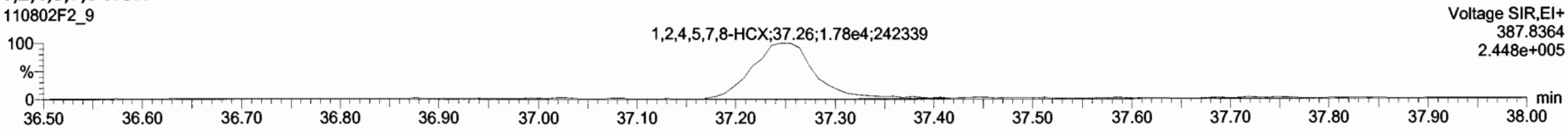
FEB 8/3/11

Dataset: Untitled

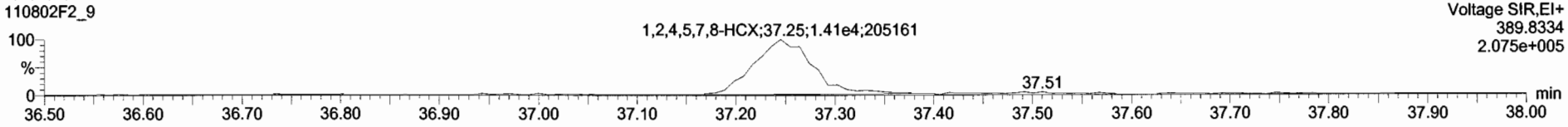
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_9, Date: 02-Aug-2011, Time: 20:54:18, ID: 27592_3922_005 MDL-AQ-5 1, Description: MDL-AQ-5

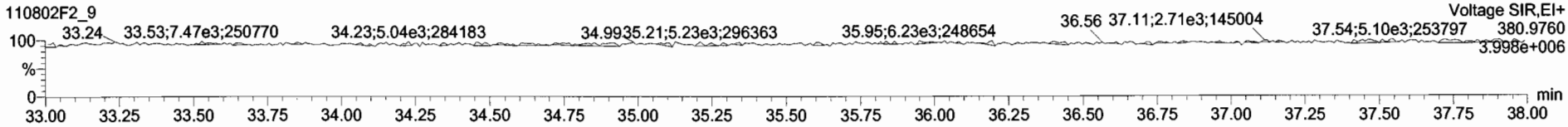
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1



TargetLynx - untitled - [Chromatogram]

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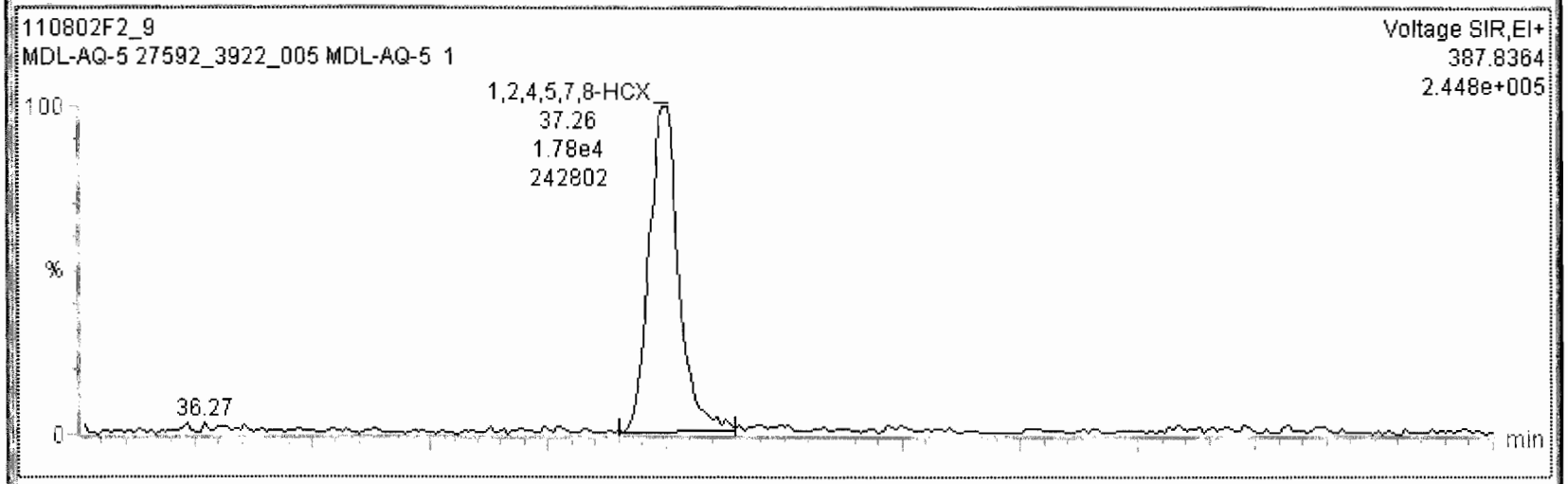
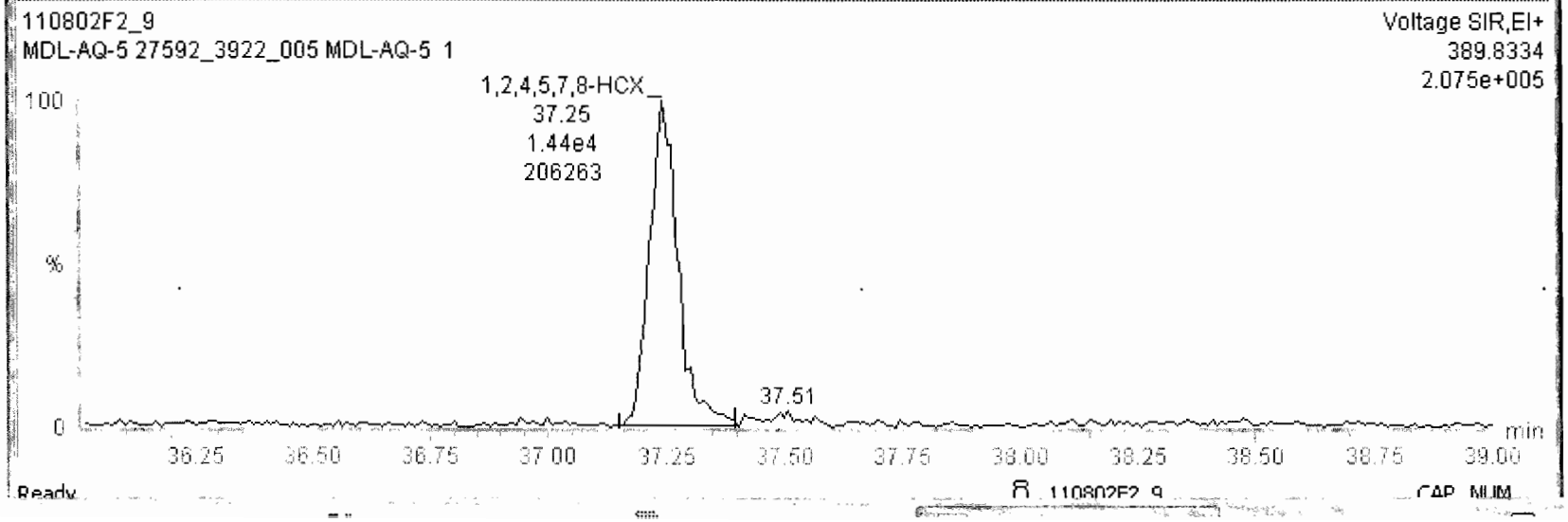
110802F2_9 - 27592_3922_005 MDL-AQ-5 1 - MDL-AQ-5

	#	Name	Resp	RA	n/y	RRF	wt/vol	RT	Conc.	%Rec	DL
1	1	1,2,4,5,7,8-HCX	3.23e4	1.23	NO	0.22	1.000	37.26	286		9.96
2	2	13C-1,2,3,7,8,9-HxCDF	1.03e6	0.52	NO	0.87	1.000	34.97	1900	94.8	3.03
3	3	13C-1,2,3,7,8,9-HxCDD	9.15e5	1.24	NO	0.72	1.000	34.55	2050	103	3.00
4	4	13C-1,2,3,4,6,9-HxCDF	1.25e6	0.53	NO	1.00	1.000	33.65	2000	100	2.63
5	5	PFK1					1.000				





Ready

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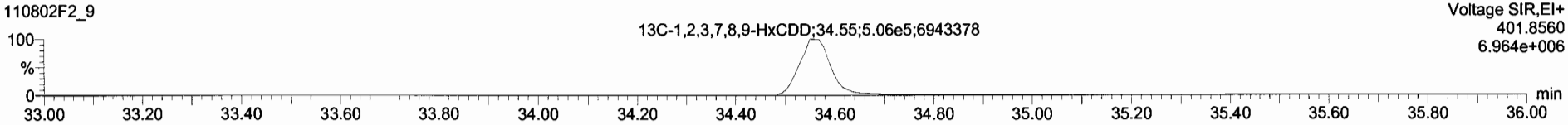
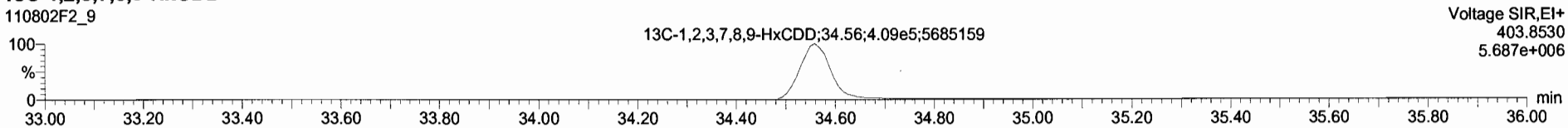
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_9, Date: 02-Aug-2011, Time: 20:54:18, ID: 27592_3922_005 MDL-AQ-5 1, Description: MDL-AQ-5

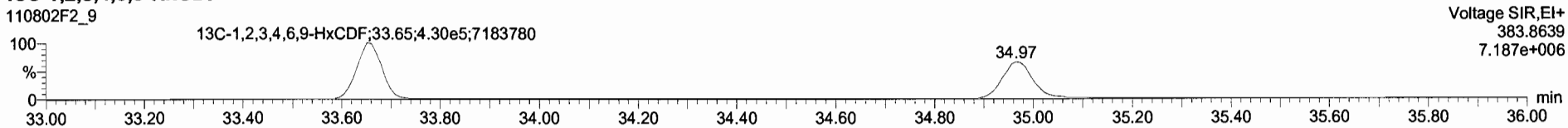
13C-1,2,3,7,8,9-HxCDD



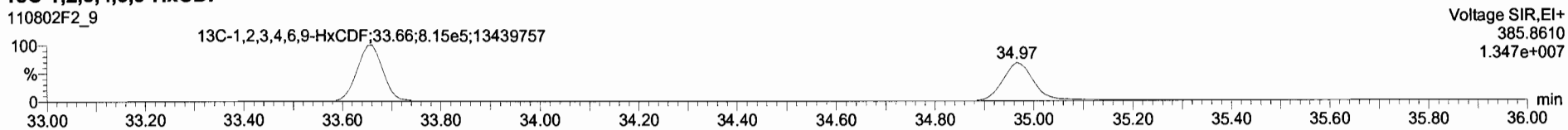
13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,4,6,9-HxCDF



13C-1,2,3,4,6,9-HxCDF



Dataset: C:\MassLynx\Default.pro\Results\110802F2\110802F2_10.qld

Last Altered: Wednesday, August 03, 2011 12:47:27 Pacific Standard Time
Printed: Wednesday, August 03, 2011 12:48:02 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-8-01-11.cdb 02 Aug 2011 09:02:25

Name: 110802F2_10, Date: 02-Aug-2011, Time: 21:37:56, ID: 27592_3922_006 MDL-AQ-6 1, Description: MDL-AQ-6

	# Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1 1,2,4,5,7,8-HCX	3.57e4	1.35	NO	0.220	1.000	37.25	315.00		9.41
2	2 13C-1,2,3,7,8,9-HxCDF	1.03e6	0.54	NO	0.869	1.000	34.98	1804.7	90.2	2.67
3	3 13C-1,2,3,7,8,9-HxCDD	9.77e5	1.24	NO	0.717	1.000	34.56	2072.9	104	3.49
4	4 13C-1,2,3,4,6,9-HxCDF	1.31e6	0.52	NO	1.00	1.000	33.66	2000.0	100	2.32

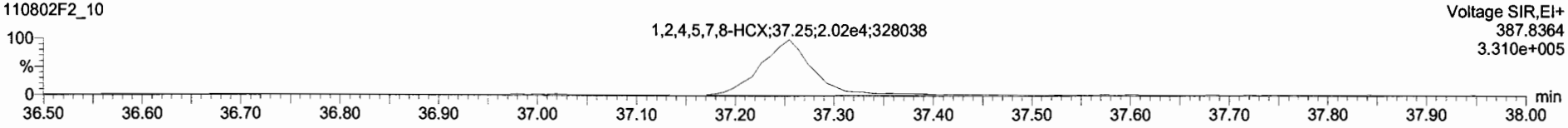
FEB 8/3/11

Dataset: Untitled

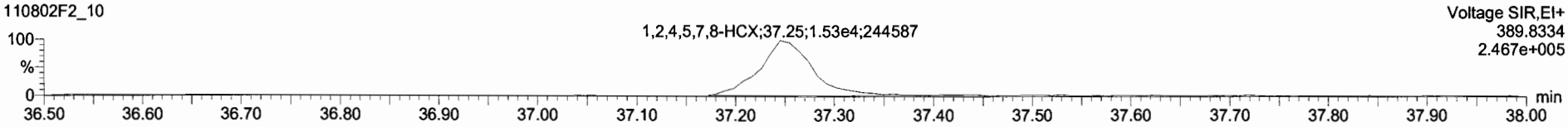
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_10, Date: 02-Aug-2011, Time: 21:37:56, ID: 27592_3922_006 MDL-AQ-6 1, Description: MDL-AQ-6

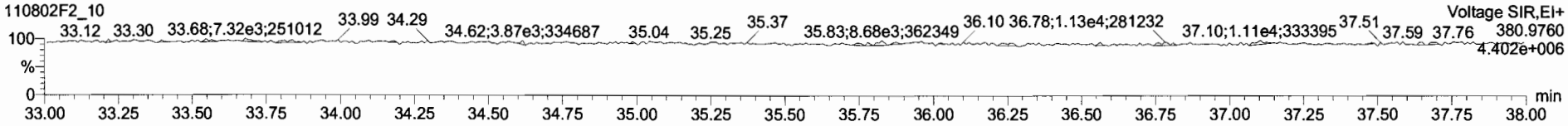
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1



TargetLynx - untitled * - [Chromatogram]

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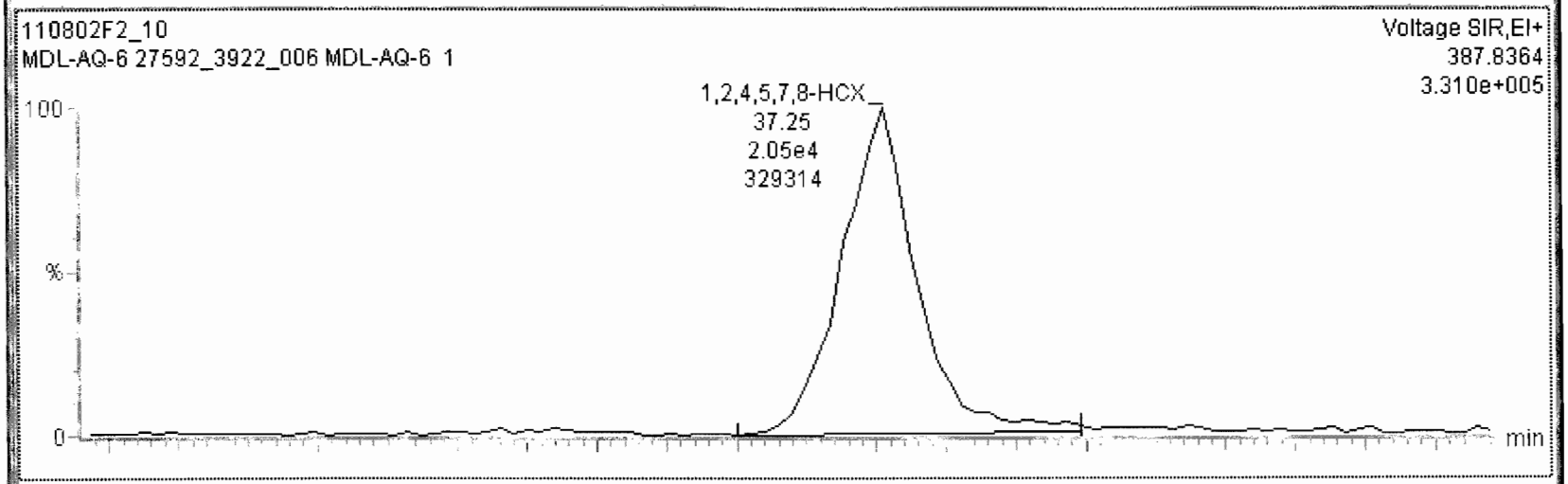
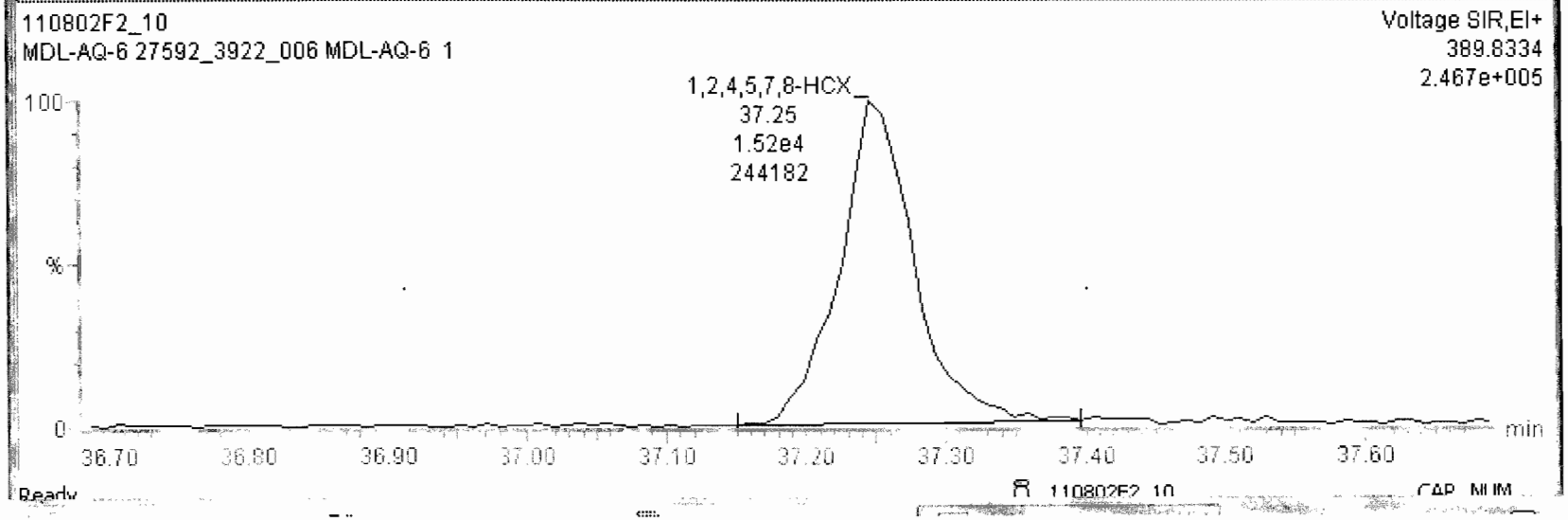
110802F2_10 - 27592_3922_006 MDL-AQ-6 1 - MDL-AQ-6

	#	Name	Resp	RA	n/y	RRF	wt/vol	RT	Conc.	%Rec	DL
1	1	1,2,4,5,7,8-HCX	3.57e4	1.35	NO	0.22	1.000	37.25	315		9.41
2	2	13C-1,2,3,7,8,9-HxCDF	1.03e6	0.54	NO	0.87	1.000	34.98	1800	90.2	2.67
3	3	13C-1,2,3,7,8,9-HxCDD	9.77e5	1.24	NO	0.72	1.000	34.56	2070	104	3.49
4	4	13C-1,2,3,4,6,9-HxCDF	1.31e6	0.52	NO	1.00	1.000	33.66	2000	100	2.32
5	5	PFK1					1.000				





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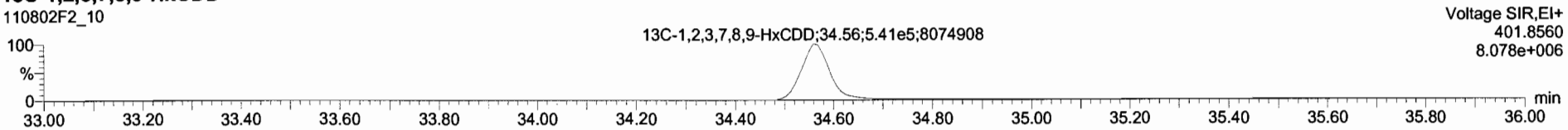
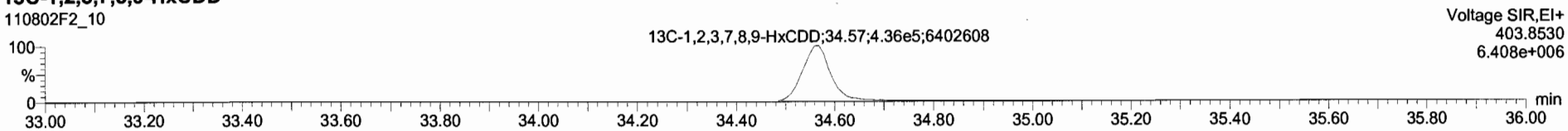
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_10, Date: 02-Aug-2011, Time: 21:37:56, ID: 27592_3922_006 MDL-AQ-6 1, Description: MDL-AQ-6

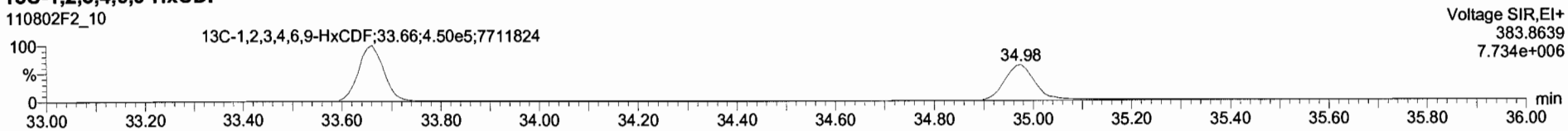
13C-1,2,3,7,8,9-HxCDD



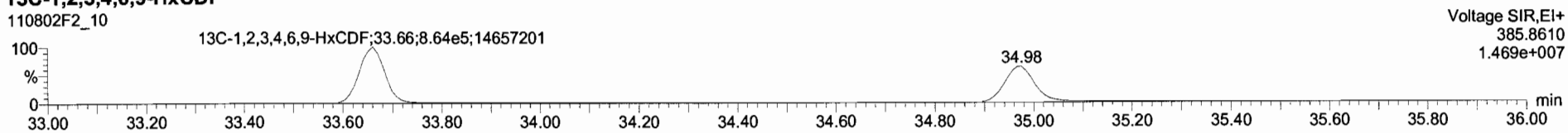
13C-1,2,3,7,8,9-HxCDD



13C-1,2,3,4,6,9-HxCDF



13C-1,2,3,4,6,9-HxCDF



Dataset: C:\MassLynx\Default.pro\Results\110802F2\110802F2_11.qld

Last Altered: Wednesday, August 03, 2011 12:50:30 Pacific Standard Time
Printed: Wednesday, August 03, 2011 12:51:12 Pacific Standard Time

Method: C:\MassLynx\DEFAULT.PRO\MethDB\hcx.mdb 27 Jul 2011 16:37:10
Calibration: C:\MassLynx\DEFAULT.PRO\CurveDB\db-5_hcxvg9-8-01-11.cdb 02 Aug 2011 09:02:25

Name: 110802F2_11, Date: 02-Aug-2011, Time: 22:21:34, ID: 27592_3922_007 MDL-AQ-7 1, Description: MDL-AQ-7

	# Name	Resp	RA	n/y	RRF M...	wt/vol	RT	Conc.	%Rec	DL
1	1 1,2,4,5,7,8-HCX	3.38e4	1.34	NO	0.220	1.000	37.25	294.15		9.00
2	2 13C-1,2,3,7,8,9-HxCDF	1.05e6	0.53	NO	0.869	1.000	34.96	1950.2	97.5	2.87
3	3 13C-1,2,3,7,8,9-HxCDD	9.66e5	1.26	NO	0.717	1.000	34.55	2182.9	109	3.20
4	4 13C-1,2,3,4,6,9-HxCDF	1.23e6	0.54	NO	1.00	1.000	33.64	2000.0	100	2.49

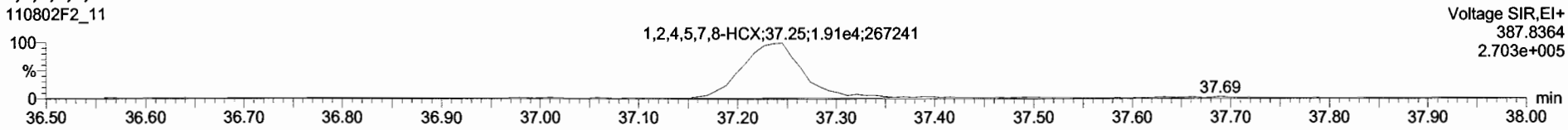
FEB 8/5/11

Dataset: Untitled

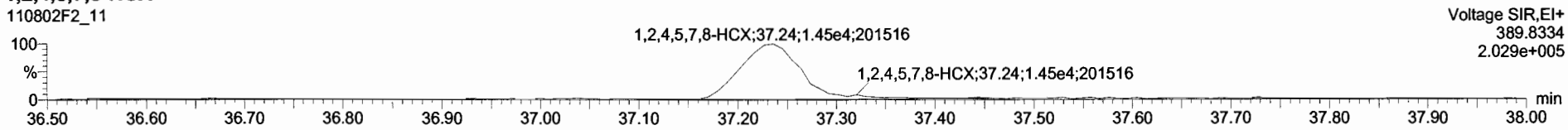
Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
Printed: Wednesday, August 03, 2011 11:37:30 Pacific Standard Time

Name: 110802F2_11, Date: 02-Aug-2011, Time: 22:21:34, ID: 27592_3922_007 MDL-AQ-7 1, Description: MDL-AQ-7

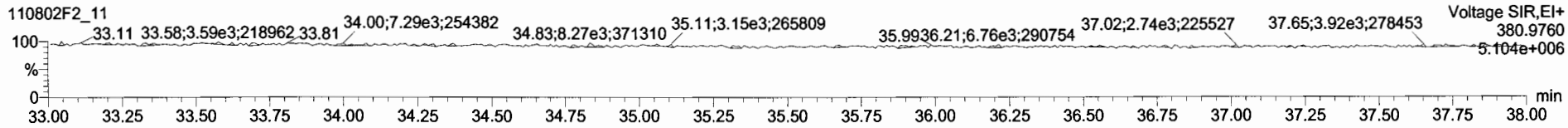
1,2,4,5,7,8-HCX



1,2,4,5,7,8-HCX



PFK1

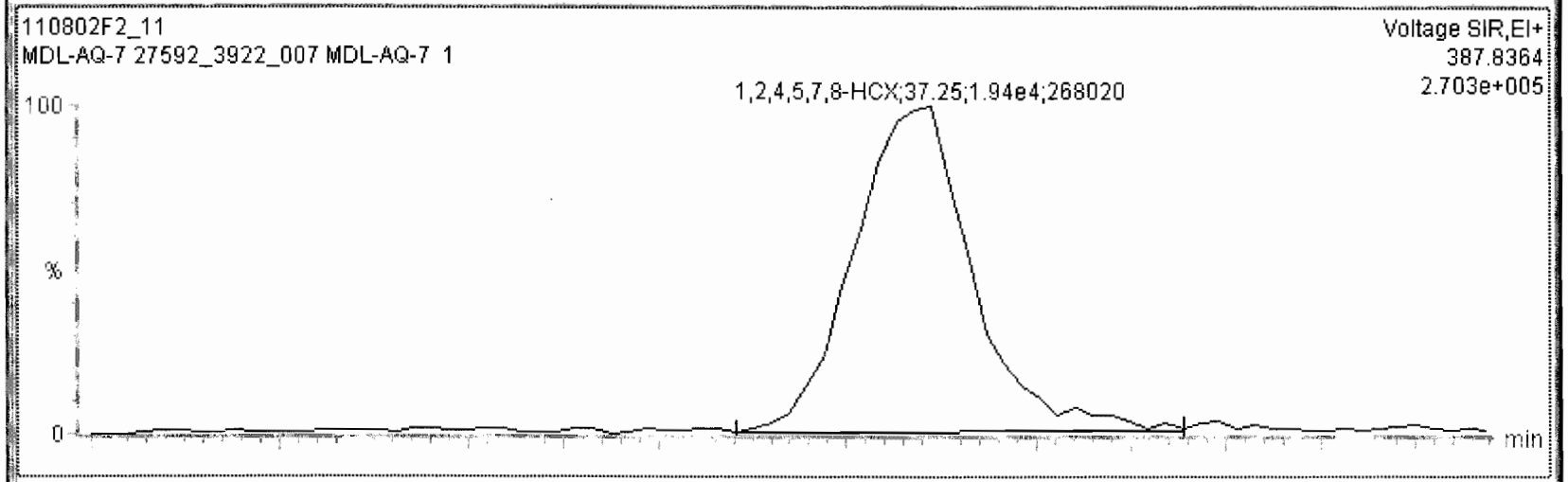


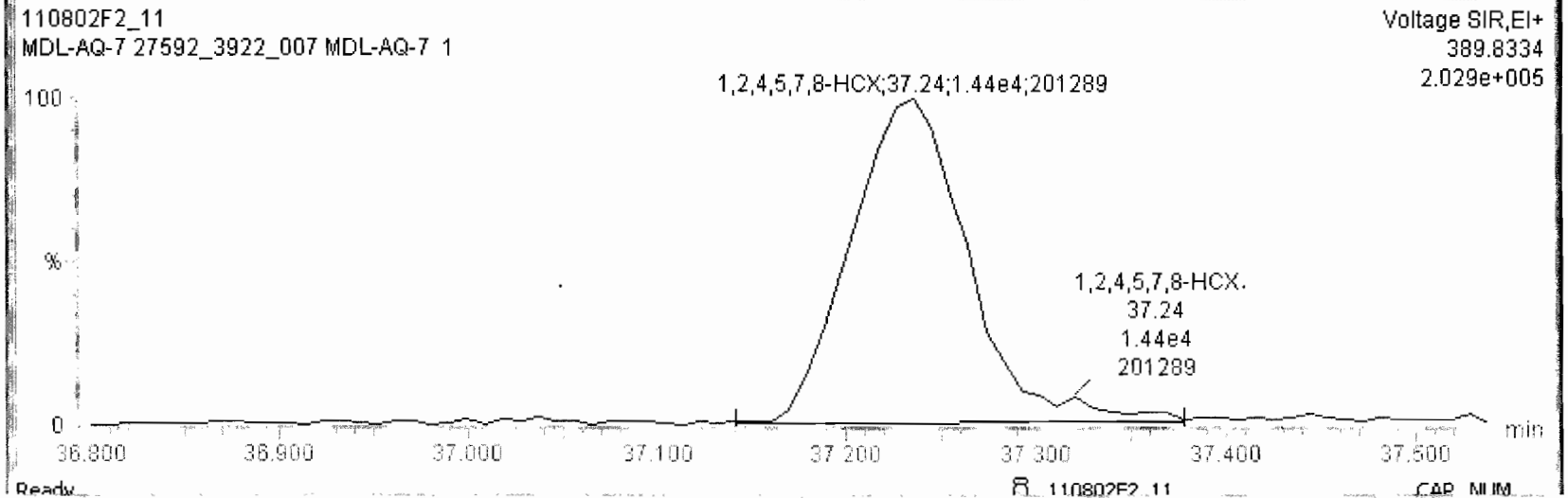
TargetLynx - Untitled - [Chromatogram]

File Edit View Display Processing Window Help

110802F2_11 - 27592_3922_007 MDL-AQ-7 1 - MDL-AQ-7

	#	Name	Resp	RA	n/y	RRF	wt/vol	RT	Conc.	%Rec	DL
1	1	1,2,4,5,7,8-HCX	3.38e4	1.34	NO	0.22	1.000	37.25	294		9.00
2	2	13C-1,2,3,7,8,9-HxCDF	1.05e6	0.53	NO	0.87	1.000	34.96	1950	97.5	2.87
3	3	13C-1,2,3,7,8,9-HxCDD	9.66e5	1.26	NO	0.72	1.000	34.55	2180	109	3.20
4	4	13C-1,2,3,4,6,9-HxCDF	1.23e6	0.54	NO	1.00	1.000	33.64	2000	100	2.49
5	5	PFK1					1.000				





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Last Altered: Wednesday, August 03, 2011 11:37:04 Pacific Standard Time
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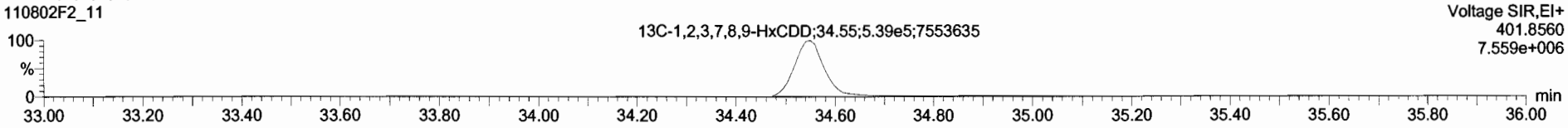
Name: 110802F2_11, Date: 02-Aug-2011, Time: 22:21:34, ID: 27592_3922_007 MDL-AQ-7 1, Description: MDL-AQ-7

13C-1,2,3,7,8,9-HxCDD



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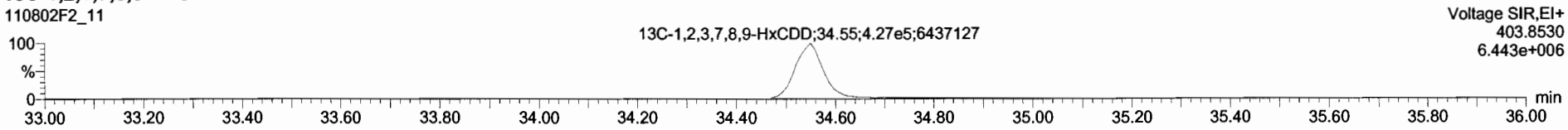

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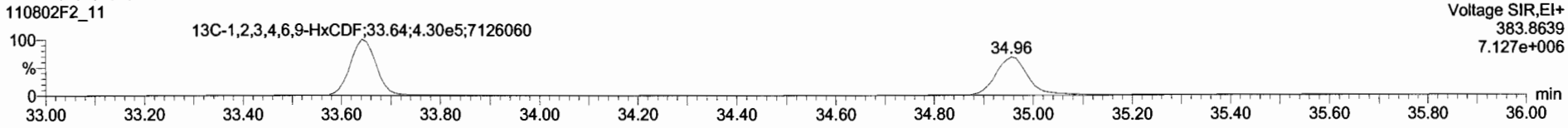

13C-1,2,3,4,6,9-HxCDF